	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 1 of 31



Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials

SBIR Phase I Final Report


2008-07-31

CONTRACT NO: **DTRT57-08-C-10022**
OUR FILE NO: **2007-DOT-003-0326**

PRIVATE INFORMATION

CONTENTS OF THIS REPORT SHALL NOT BE DISCLOSED WITHOUT THE CONSENT OF INTEGRAN TECHNOLOGIES U.S.A. INC.

Integran Technologies U.S.A. Inc., 2541 Appletree Dr., Pittsburgh, PA 15241, USA.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 2 of 31

Executive Summary

A DoT-funded 6-month Phase I SBIR preliminary feasibility study aimed at investigating whether electroformed high strength nanocrystalline metal sleeves can be employed as steel pipeline Crack Arrestors (CA's) was carried out. The DoT objective is to support the development of a technology capable of arresting pipeline running fractures *in a soft manner* and without “ring-off” i.e. to arrest the crack within the CA and without complete circumferential catastrophic fracture of the pipe. This need is particularly acute as high strength steels, which may not have sufficient intrinsic material toughness to overcome running fractures, continue to proliferate in the transportation of high-energy rich natural gas, CO₂, etc.

It was originally proposed that Nanometal electrodeposition is a good candidate for such an application because the material exhibits an excellent combination of high strength and toughness and can be readily applied to the outer pipe surface with a non-uniform thickness distribution tailored specifically to stop the crack more effectively *within the CA* as opposed to the CA edge. In addition, the process to electrodeposit structural layers of nanometal has already been ASME-approved and industrially implemented for the in-situ crack repair of degraded steam generator tubes in the nuclear industry and is therefore a proven process piping repair technology.

In order to judge the feasibility of the concept, 3 key Tasks were performed in Phase I:

1. Technology Review with pipeline industry personnel
2. Lab work to demonstrate that Nanometal can be readily applied to X100 pipeline steel
3. Finite Element (FE) Modeling of the crack arrest effectiveness of the monolithic Nanometal-based designs – carried out by Centro Sviluppo Materiali (CSM), an acknowledged world leader in the simulation of pipeline crack arrest

The completion of these Tasks resulted in the following general conclusions being drawn:

1. FE Simulations revealed that X100 steel pipeline running fracture arrest can be successfully achieved with Nanometal-based CA's
2. The Nanometal CA thickness required to arrest the propagating crack will likely be approximately 6mm. Application of 6mm of Nanometal would likely require ~20hrs to apply *in situ*. The industry experts pointed out that 20hrs is not practical for an in-field CA installation.
3. Any workable Nanometal-based CA design must therefore be one that is pre-fabricated in advance of in-field installation as opposed to electrodeposited directly onto the pipe surface *in situ*.
4. It is proposed that the technical, economic and practical in-field installation feasibility objectives can be met with a design comprised of Nanometal foil wrapped tightly around the pipe OD in a fashion similar to the well-known Clockspring™ CA technology, potentially including high energy absorption polyurethane-based binding interlayers.
5. It is proposed that the development of this “wrapped Nanometal foil” concept comprise the central focus of Phase II activity, and that any prototype designs be evaluated in-field and via FE simulations in continued collaboration with CSM.



	<p align="center">Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report</p>		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 3 of 31

Table of Contents

1.0	INTRODUCTION.....	4
1.1	Crack Arrest Background and Introduction to the Nanometal-Based Concept.....	4
1.2	Background - Electrodeposited Nanostructured Materials For Process Pipe Repair	5
1.2.1	Synthesis & Structure of Electrodeposited Nanoscale Metals and Alloys	5
1.2.2	Strength and Toughness of the Nanometal Material	5
2.0	PHASE I TECHNICAL OBJECTIVES	7
3.0	PHASE I RESULTS.....	8
3.1	Task 1 – Technology Review.....	8
3.1.1	Recommendations of the Industry Advisory Group:.....	9
3.1.2	Task 1 Conclusions	9
3.2	Task 2 – Laboratory Proof of Concept.....	9
3.2.1	Test Sample Production.....	9
3.2.2	Deposit Characterization	10
3.2.3	Mechanical Testing	11
3.2.4	Adhesion Testing.....	14
3.2.5	Task 2 Conclusions	16
3.3	Task 3 – Finite Element (FE) Simulations	16
3.3.1	Introduction	16
3.3.2	Preliminary Calculations	16
3.3.3	Introduction to CSM’s PICPRO® Code.....	17
3.3.4	Introduction to CSM’s Nanometal CA Simulations	19
3.3.5	Results of CSM Finite Element Simulations	20
3.3.6	Some Comments on “Soft” vs. “Hard” Arrest.....	22
3.3.7	Task 3 Conclusions	24
3.4	Overall Phase I Conclusions	24
3.4.1	Technical Feasibility	24
3.4.2	Practical In-Field CA Installation Feasibility	24
3.4.3	Economic Feasibility	25
4.0	PHASE II ACTIVITIES.....	26
4.1	Task 1 – Development of Nanometal-Enabled CA Design.....	26
4.2	Task 2 - Modification of CSM Code to Guide CA Design	29
4.3	Task 3 - Larger Scale Burst Testing.....	29
5.0	REFERENCES.....	30
6.0	APPENDIX A – Final Report from Centro Sviluppo Materiali S.p.A.: “Adoption of Nanostructured Metal Alloy in Crack Arrestor Design – Feasibility Study by FEM Analysis”	31

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 4 of 31

1.0 INTRODUCTION

1.1 Crack Arrest Background and Introduction to the Nanometal-Based Concept

Crack arrest of a ductile rupture is a major issue for high-energy (rich gas, high-pressure, CO₂) pipelines, especially those constructed from newer high-strength (X-100, X-120) and/or low-toughness steels^{1,2,3,4}. Crack initiation could result from either an environmentally assisted crack (e.g., a stress corrosion crack) or internal or external localized corrosion.


Various crack arrestor designs have been proposed and/or implemented on pipeline systems¹⁻⁴, including: light-weight fiber wraps (e.g., ClockSpring™ or Composite Reinforced Linepipe), steel sleeves (tight, loose, and grouted), and welded or clamped rings. Typically, these crack arrestors would be installed in the field either during or following laying of the pipe, resulting in an additional stage in construction or the need to excavate following construction.

An additional consideration is the effectiveness of the crack arrestor. Loose-fitting steel sleeves may not stop the propagating crack, whereas tight sleeves can result in such effective crack arrest that “ring-off” or “hard arrest” (circumferential deflection) of the crack occurs resulting in possible ejection of the pipe from the ground⁴. A “soft arrest”, in which the crack propagates through and partially beyond the crack arrestor, is preferable⁴.

The objective of this 6-month Phase I effort was to explore a new application of an existing technology as a novel crack arrestor tool for high-strength pipelines. The full-encirclement crack arrestor would comprise a thin layer or “sleeve” of electrodeposited nanocrystalline material applied to the outside of the pipe either in the coating mill during fabrication or in the field during or following construction e.g. applied to localized regions known to be prone to failure, retrofitted, etc. Mill application would be performed during the coating stage, after surface preparation and before application of the mainline coating.

There are several key features of this technology that could potentially make it an excellent match with current DoT needs:

1. the Nanometal-based tubing repair concept is mature, having been developed for the nuclear industry in the early/mid 1990’s;
2. Integran is already focused on the adaptation of the process to (albeit smaller diameter) oil & gas piping and so the technical risks involved in pipe Nanoplatting are relatively low;
3. because of the small grain size, the material exhibits exceptional yield and tensile strengths (5-10x that of conventional grain-size material) without significantly sacrificing ductility. Because of the higher strength, only a thin layer of electrodeposit would be required;
4. in the scenario where the nanometal is deposited directly onto the steel pipe surface, the nanometal reinforcement layer has been proven to possess a level of adhesion to steel that is stronger than the yield strength of many common steels themselves and so sleeve disbonding is rarely a concern;
5. the Young’s modulus of the nanometal reinforcement layer is nearly the same as that of steel, so there will be no “stiffness mismatch” between the mechanical crack arrestor sleeve and the steel pipe material;
6. the nanometal reinforcement layer is nickel- or cobalt-based and so its corrosion resistance is

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 5 of 31

excellent;

7. the nanocrystalline metal that is applied by the process is well-characterized, ASME-approved, and possesses a microstructure that has been designed specifically to exhibit a combination of high strength and good ductility that make it ideally suited for application as a soft crack arrestor where excellent material toughness is required.
8. because the strength of the deposit can be finely controlled through either the thickness and/or grain size of the electrodeposit, the properties of the crack arrestor could be matched to the properties of the pipe (grade and wall thickness) and the service conditions (nature of the gas, pressure). Such a crack arrestor technology would be more flexible than existing technologies.

1.2 Background - Electrodeposited Nanostructured Materials For Process Pipe Repair

1.2.1 Synthesis & Structure of Electrodeposited Nanoscale Metals and Alloys

Nanostructured materials can provide a unique combination of strength and hardness with good ductility that cannot be obtained in conventional coarse-grained polycrystalline materials. This unique and novel combination of properties is a direct result of the ultrafine-grained structure of these nanocrystalline materials (see Figure 1 below). In addition, electrodeposition is the simplest and most cost-effective method for producing nanostructured materials. Consequently, because of well-established electroplating practices as well as mature infrastructure, electrodeposited nanostructured materials have rapidly advanced to a number of commercial applications. In the late 1980's, Integran's principals pioneered the earliest research and development studies on the use of electrodeposition to produce nanocrystalline materials^{5,6}. Further they were the first to demonstrate the benefits and versatility of nanostructured metals in an actual application (the ElectrosleeveTM process for nuclear steam generator repair, 1993)^{7,8,9} and they also own some of the earliest issued US patents in the field of nanotechnology; the general conditions for producing nanocrystalline metals and alloys by electrodeposition are documented in US Patent Nos. 5,352,266 (Oct.4, 1994) and 5,433,797 (July 18, 1995)^{10,11}.

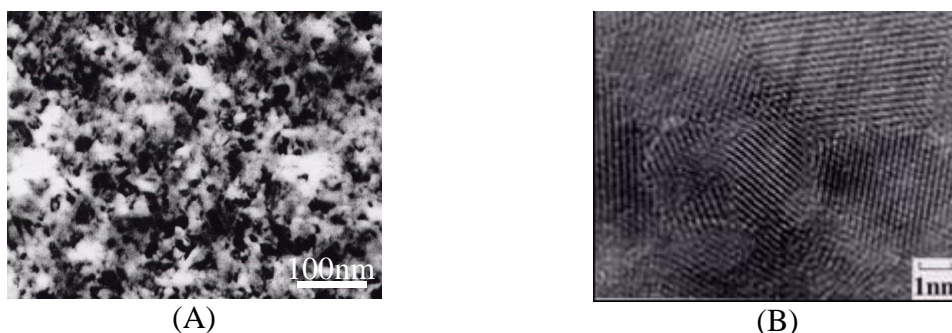



Figure 1 (A) transmission electron micrograph (TEM) of electroformed nanocrystalline Ni, (B) high resolution micrograph of electroformed nanocrystalline Ni .

1.2.2 Strength and Toughness of the Nanometal Material

As a result of Hall-Petch grain size strengthening, nanocrystalline materials display significant increases in hardness and strength relative to their coarser-grained counterparts, which render them ideally suited for applications requiring high strength for structural reinforcement purposes. When compared to their

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 6 of 31

conventional counterparts, 4 to 7-fold increases in hardness and 2 to 4-fold increases in tensile strength are typically observed, yet the materials remain stable to quite high temperatures. Figure 2(a) shows the effect of grain size on the yield strength of pure nickel, showing the significant increase in yield strength when the average grain size is decreased below 100nm¹².

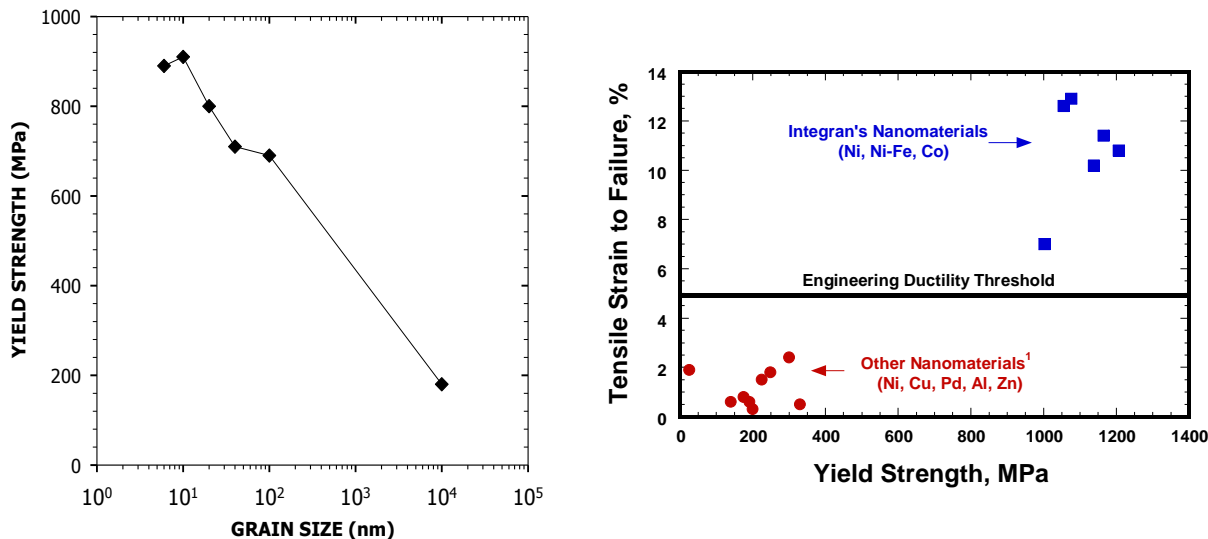



Figure 2 (a) Yield strength of pure nickel as a function of its grain size¹²; (b) Tensile strain to failure versus yield strength for various nanomaterials¹³.

Even with the substantial enhancements in strength and hardness, Integrant's nanocrystalline metals and alloys have been found to have excellent toughness and ductility. This is in contrast to nanometals prepared by other synthesis techniques (such as powder consolidation, etc) with which the ductility has been shown to decrease rapidly with decreasing grain size to levels less than 3%¹³. In many cases, this is a result of residual porosity in the material. The difference in ductility between Integrant's nanometals and those synthesized by other technique is shown in Figure 2(b). The combination of high strength and high ductility achievable with Integrant's nanometals is quite unique and has not been matched with nanostructured materials produced by any other means¹³.

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 7 of 31

2.0 PHASE I TECHNICAL OBJECTIVES

It was originally proposed that the overall program be configured as follows:


Phase I: Proof of Concept / Feasibility Study

1. Technology Review: this would essentially comprise a review of Nanoplate® technology, its prior applications, and its applications in the pipeline industry. The objective would be to solicit feedback from pipeline operators and CA implementers in order to judge the overall technical, practical, and economic feasibility of a nanometal-based pipeline CA.
2. Laboratory Proof of Concept:
 - Demonstration of electrodeposition on sand/shot-blasted X100 surfaces
 - Testing of resulting strength of line pipe
 - Characterization of interfacial bonding between pipe steel and electrodeposit
3. Finite Element Modelling of Design of Crack Arrestors: modeling the effect of crack arrestors in order to guide the optimum design(s). This task was carried out in collaboration with Centro Sviluppo Materiali (CSM), an acknowledged world leader in pipeline crack arrestor simulation and design.

The objective of Phase I was to formulate a preliminary assessment of whether Integran's knowledge of tubing repair can be applied to the soft crack arrest of the large-diameter gas transmission pipe grades and geometries that are of specific interest to the DoT.

Phase II: Full-Scale Tests, Design Specification and Tooling Development

1. Development of Design Specification: optimum nanomaterial microstructure, coating thickness and width for a range of applications.
2. Full-Scale Tests: full-scale burst tests would be performed on various pipe samples with actual or simulated defects, including: internal and external cracking, internal and external corrosion.
3. Tooling Development: design and fabrication of the plating cells required to deposit the nanomaterial onto the pipe grades and geometries that are of interest to the DoT.

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 8 of 31

3.0 PHASE I RESULTS

3.1 Task 1 – Technology Review


The first step in the program was to engage experts from the pipeline industry in discussions regarding the technical and economic feasibility of the proposed Nanometal-based CA concept.

Industry Advisory Group Members:

1. Fraser King, Integrity Corrosion Consulting Ltd. (advisor to Integran and link to pipeline industry personnel)
2. David Horsley, BP (pipeline operator / CA implementer)
3. Millan Sen, TransCanada Pipelines (pipeline operator / CA implementer)
4. Peter Singh, BrederoShaw (pipeline coater)

Dialogue with these pipeline industry personnel resulted in the following opinions being put forward:

1. 6-month Phase I = not enough time to examine the characteristics of Nanometal sleeving with respect to its unique “crack bridging” and “*in situ* structural reinforcement and repair of degraded sections of critical process piping” for which the technology has been successfully demonstrated by Integran to be a very effective repair method for degraded heat exchanger tubing in nuclear power plants. Therefore the principal focus of this Phase I program should be on the application of Nanometal as a soft crack arrestor (CA) tool, while deferring the crack bridging/*in situ* repair potential of the Nanometal sleeving to a later stage of technology development.
2. The time required to apply a monolithic Nanometal-based CA *in situ* (onto the pipe surface in-field) is directly proportional to the Nanometal thickness required to arrest a fast running shear fracture in a natural gas-carrying pipeline made of X-100 steel. Both the practical and economic feasibility of the concept seem to be predicated upon the establishment of an estimated thickness value for the Nanometal sleeve. For example, if 6mm of Nanometal thickness (>20hrs of plating time) is required to stop the crack, then the CA sleeve cannot be electroformed *in situ* on the pipe OD because 20hrs of installation time is simply far too lengthy to be practical.
3. There are a number of very unique features of the Nanometal-based concept that could have a significant impact on its effectiveness as a CA and/or on the mechanics of crack arrest (i.e. “soft” vs “hard” arrest) that cannot be demonstrated within the auspices of this Phase I project; some of these features should therefore be fully addressed and demonstrated as part of the follow-on Phase II project. Among some of these attractive features include:
 - The ability to process the nanometal into unique shapes that are not readily possible with conventional CA fabrication methods e.g. the ability to “grade” the nanometal sleeve thickness i.e. apply a “bell shaped” cross section CA with a thick central section that would then taper off in a thinner cross section near the sleeve edge; the ability to create a Clock Spring™-style CA design with ultra-high strength/ductility/toughness Nanometal foils or Nanometal-coated fibers. This could have a significant impact on the mechanics of the “hard” vs. the safer and more effective “soft” CA crack stopping mode.

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 9 of 31

- The excellent bond strength of the nanometal compared to that of a high strength steel. This could potentially result in a more efficient crack energy dissipation /distribution mechanism.
- The ability to apply the Nanometal *in situ*, resulting in an intrinsic compressive stress to “squeeze” the X-100 steel pipe. This may favorably impact on the dynamics of the nanometal crack arrestor sleeve.

3.1.1 Recommendations of the Industry Advisory Group:

1. Since burst testing is beyond the scope of Phase I, perform as trustworthy a FE Simulation (Task 3) as possible and obtain a “best guess” monolithic Nanometal CA thickness estimate from these CA simulations
2. Estimate the technical, practical installation, and economic feasibility of the overall concept on the basis of how much Nanometal is required and how long it takes to apply in-field

3.1.2 Task 1 Conclusions


In conclusion, dialogue with pipeline operators and CA implementers has proved to be extremely helpful. Specifically, it was on the basis of the recommendation of the Industry Advisory Group that we established a successful working relationship with Centro Sviluppo Materiali (CSM), an acknowledged world leader in pipeline crack arrestor simulation and design. This proved to be an extremely valuable interaction (see Task 3 below) and we anticipate that the probability of success in Phase II has been significantly improved owing to CSM’s experience and expertise that we should be able to leverage as the design process continues. Perhaps more importantly, the Industry Advisory Group provided us with the context for what would be required to make the Nanometal-based CA design a success: not only must it stop running fractures in high strength steel in a “soft” manner, but it must be suitable for (ideally, in-field) installation that can be carried out within a reasonable period of time (~2-6 hrs) and at a reasonable cost (<\$10-20k per CA).

3.2 Task 2 – Laboratory Proof of Concept

The goal of this task was to gather technical data to confirm Integran’s previous experience that high strength nanostructured metal can be deposited onto high strength steel pipe and that it affords a considerable increase in the strength of the pipe. It is anticipated that the high strength Nanometal layer will ultimately result in a significant decrease in the propagation rate of cracks in the high strength steel, though the full-scale confirmation of this assumption was beyond the scope of Phase I testing.

3.2.1 Test Sample Production

Through our Industry Advisory Group activities, we were able to identify a source for X100 pipeline steel. A ¼ section of pipe was procured and sections cut into samples appropriate for Nanometal electrodeposition. The majority of the X100 substrate samples were 4” x 0.5” x 0.0625” strips onto which Nanometal deposition optimization trials were carried out. As the most important feature of the Nanometal-based CA material is its high strength, the strongest Integran alloy available (Co-P) was selected as the material of choice for the majority of the Task 2 activities. However, some nanocrystalline Ni and Ni-Fe samples were also subjected to mechanical property characterization, most notably the high

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 10 of 31

strain rate testing outlined below. The coating thickness of the Nanometal layers on X100 was varied between 0.1 and 1mm and most samples were processed with the standard Integran nanocrystalline CoP electrodeposition process using the tank shown in Figure 3 below.



Figure 3 Photograph of the Nanometal electroplating cell used to process the majority of the samples investigated in this Phase I study.

3.2.2 Deposit Characterization

For the nanocrystalline materials deposited throughout this study, care was taken to ensure that the target microstructure was consistently achieved. Figure 4 below contains an optical micrograph and an X-Ray Diffraction (XRD) pattern taken from a typical Nanometal deposit. The “cauliflower” surface morphology of the Nanometal is similar to that typically observed for ultrafine-grained electrodeposits. In addition, the XRD profile exhibits significant peak broadening, the extent of which indicates that the average grain size of this particular CoP Nanometal coating material was approximately 5-15 nm in diameter.

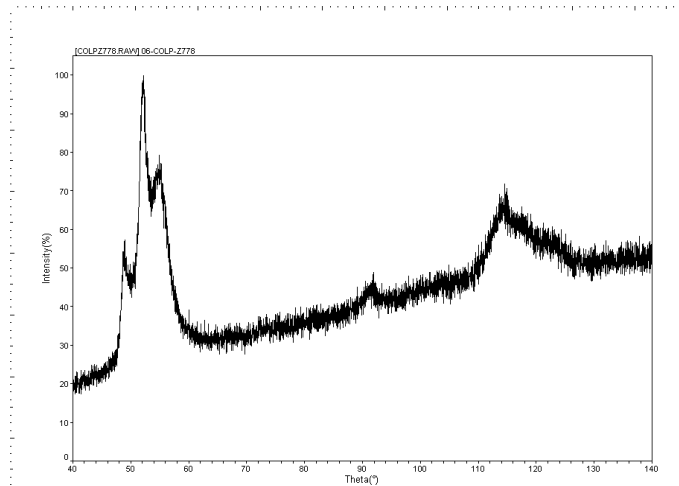
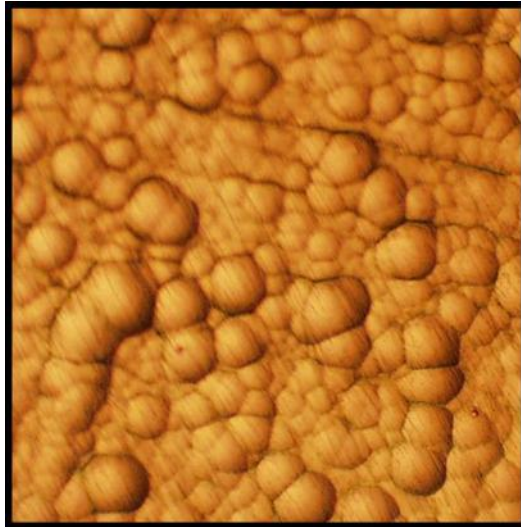


Figure 4 (a) Surface of the Nanometal coating material showing the “cauliflower” surface morphology that is typical of nanocrystalline electrodeposits; (b) X-Ray Diffraction (XRD) pattern for the Nanometal coating material showing significant peak broadening originating from extreme grain refinement (grain size: 5-15 nm).

3.2.3 Mechanical Testing


The objective of this sub-task was to perform mechanical testing of the Nanometal and compare it to the X100 pipeline steel benchmark material. This was done to demonstrate that significant structural reinforcement of the pipeline structure can be achieved with a relatively thin sleeve of Nanometal.

3.2.3.1 Hardness Testing

The Vickers hardness of the X100 pipeline steel in the as-received condition was measured to be 278 VHN. Using the standard $H_V = 3 \cdot \sigma_{UTS}$ hardness-strength approximation, this translates into a tensile strength of 915 MPa or 130 ksi, which is near the expected value for X100 steel (“100” refers to the specified minimum yield strength as opposed to the ultimate tensile strength). In contrast, the nanocrystalline CoP coating exhibited a much higher hardness value between 530-600 VHN. This hardness value yields a tensile strength estimate of 1730-1960 MPa or 250-280 ksi. This tensile strength estimate is consistent with values obtained using monolithic plates of CoP material previously tensile tested at Integrant. In summary, hardness testing indicates that the Nanometal is twice as strong as the X100 steel. This was subsequently confirmed by tensile testing of each material, the results of which can be found in Figure 5(a) below.

3.2.3.2 Quasi-Static Tensile Testing

Round tensile bars conforming to the ASTM E8-01 “Standard Test Methods for Tension Testing of Metallic Materials” specification were machined from the X100 pipeline steel. Samples were coated with nanocrystalline CoP to a thickness of approximately 0.16 mm (0.0065”). This resulted in an increase in the gauge section diameter of the steel tensile bars from 6.2 mm (uncoated) to 6.5 mm (coated). Quasi-static tensile testing of the uncoated and coated specimens yielded the curves contained in Figure 5(b)

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 12 of 31

below. For reference, the tensile curves for the standalone X100 and monolithic samples of Nanometal are contained in Figure 5(a) – note the change in axes between Figures 5(a) and (b).

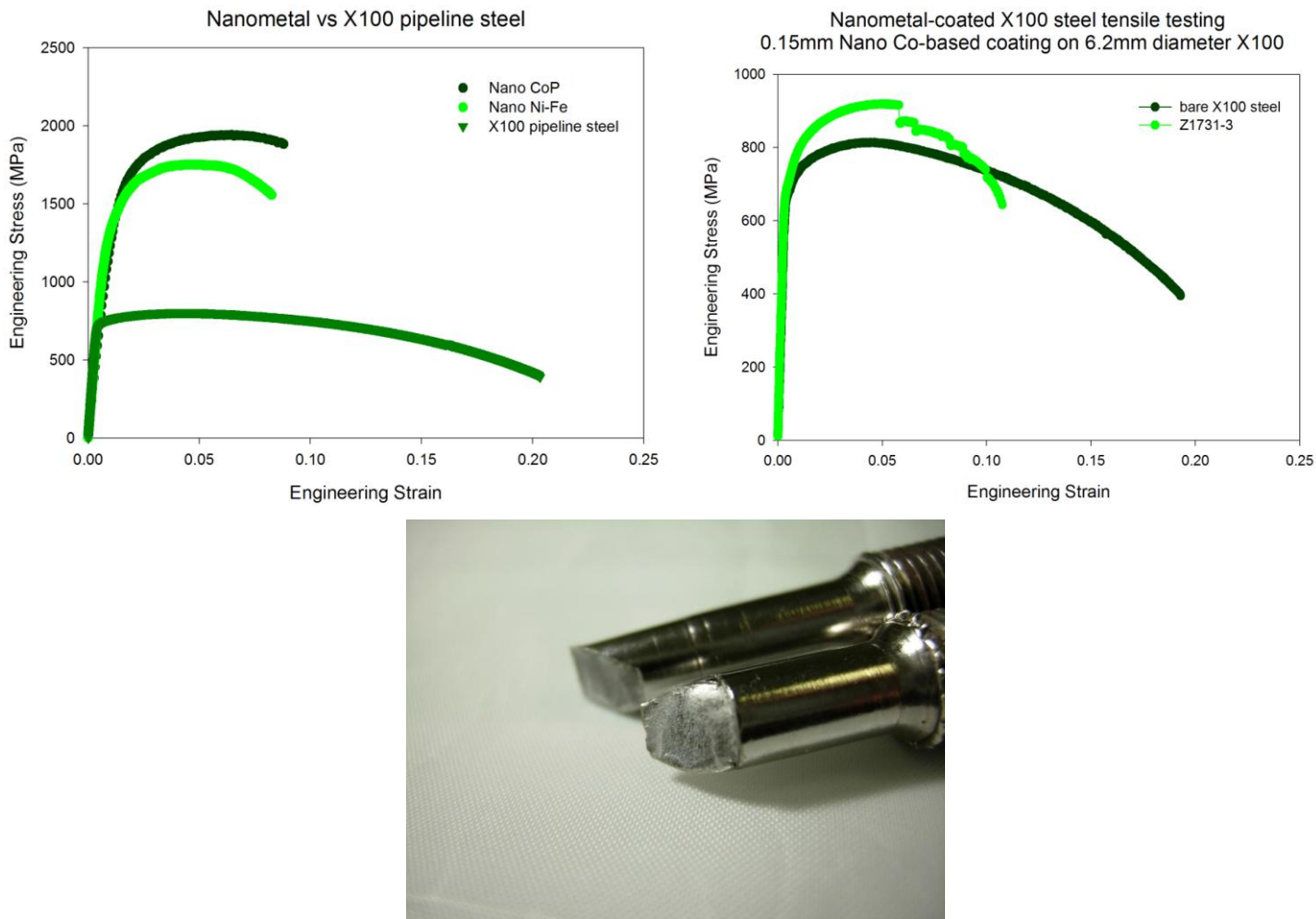



Figure 5 (a) Quasi-static tensile engineering stress-strain curves of standalone X100 steel and standalone Nanometal; (b) quasi-static tensile engineering stress-strain curves of uncoated and Nanometal-coated X100 pipeline steel; (c) photograph of a fractured specimen.

It can be seen from Figure 5(b) that a significant improvement in the strength of the line pipe material was imparted by the Nanometal coating even though it comprised only 9% of the cross-sectional area of the tensile specimen. In addition, because of the strengthening effect imparted by the Nanometal (twice as strong but half the ductility vs. X100), the overall structure was able to sustain a much higher applied load as compared to bare X100 steel but once the less ductile coating fractured, this higher load was immediately transferred to the steel and the sample broke soon thereafter. Given the properties of each of the constituent metals (Figure 5(a)), this behavior is to be expected. It is important to note that this effect does not imply that the Nanometal CA could represent a mechanical debit to the steel pipe, but simply

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 13 of 31

reflects the fact that the Nanometal is half as ductile as the X100 steel and therefore is unable to sustain as much plastic deformation as X100 steel. Indeed, as was demonstrated in Task 3 (see Section 3.3 below), the 2x strength of the Nanometal can successfully contribute to a fully functional CA material, as long as its own ductility threshold is not surpassed (ductility was accounted for in all the simulations).

Figure 5(c) contains a photograph of a coated tensile specimen taken after testing. The effective load transfer between coating and substrate is reflected in the overall compatibility of deformation of the two materials coupled with the observation that the coating did not delaminate from the X100 substrate (see Section 3.2.4 below for a more detailed description of this feature).

3.2.3.3 High Strain Rate Kolsky Bar Testing

The original Phase I Workplan called for quasi-static tensile and bond strength testing of the proposed monolithic nanometal-on-steel geometry. It has since been appreciated to a much greater extent that the pipeline running fracture scenario is an extremely energetic, high strain rate event that may call into doubt the validity of quasi-static mechanical testing on its own. This resulted, in part, from an interaction with Tom Siewert, Deputy Chief of Pipeline Reliability at NIST, who encouraged us to pursue high strain rate mechanical testing on the Integran nanocrystalline metals and alloys. The Kolsky bar tests provide compressive stress-strain information at strain rates of the order of $\sim 3000 \text{ s}^{-1}$, which is 7 orders of magnitude greater than the conventional quasi-static strain rate stress-strain testing (normally conducted in the range of $\sim 5 \times 10^{-4} \text{ s}^{-1}$). The objective of the Kolsky bar mechanical test is to gain valuable information and insight on the intrinsic toughness of the nanometal sleeve, as compared to that of the high strength pipeline X-100 steel, at very high rates of deformation. This scenario is much more applicable to a pipeline fast running shear fracture and together with the response of the nanometal crack arrestor in first slowing down and afterwards eventually stopping the fast moving crack. To this end, monolithic X100 and Nanometal sample coupons were prepared and tested by Dr. Steven Mates at NIST. Figure 6 below contains the main results. Unfortunately, due to practical electroforming issues, it was not possible to fabricate a Co-based sample of appropriate shape for the Kolsky bar test, and so the results shown are for Ni-based nanocrystalline materials only. Given their similar quasi-static tensile curves, it is anticipated that the performance of the Co-P would be quite similar to the Ni-Fe shown in Figure 6.

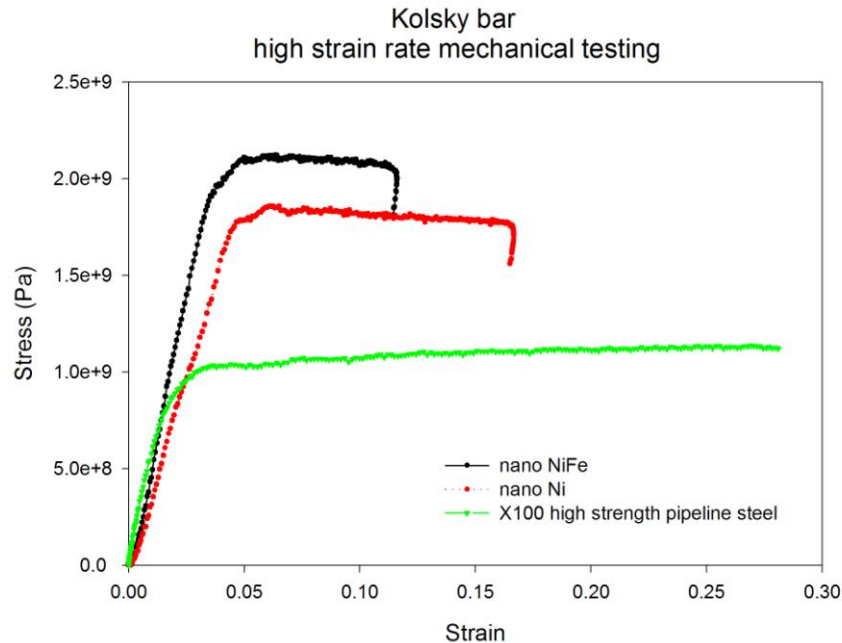



Figure 6 Results of high strain rate ($\sim 3000 \text{ s}^{-1}$) Kolsky bar testing of X100 pipeline steel as compared to electrodeposited Nanometal, demonstrating that the significant strength difference between the two materials is preserved at high rates of deformation. Courtesy of Dr. Steven Mates, NIST.

As was the case in quasi-static mechanical testing, it can be seen from Figure 6 that the Nanometal is approximately 1.8 – 2x as strong as the X100 pipeline steel under Kolsky bar loading conditions. In addition, the Nanometals tested exhibited good ductility and toughness under high strain rate loading. The implication is that, being nearly twice as strong as steel, renders the Nanometal capable of imparting a significant structural reinforcement effect to high strength pipeline steel such as would be required locally in the crack arrestor scenario.

3.2.4 Adhesion Testing

3.2.4.1 Introductory Comments

It should be noted that the question of whether good adhesion of Nanometal to the steel substrate is a critical CA design parameter at all has been a matter of some debate throughout the program. Initially, it was thought that more effective load transfer might be accommodated by ensuring a high bond strength value between the Nanometal and the steel via electrochemical cleaning and highly adhesive Nanometal electroforming directly onto the pipe surface. On the other hand, conversations with the Industry Advisory Group and CSM have indicated that, while the CA must certainly be in close mechanical contact with the pipe outer surface, the impact of an electrochemically “activated” bond vs. a tight mechanical fit may be negligible. This view has been based upon the results of FE simulations (CSM) and practical in-field CA performance experience with conventional composite-based CA’s which are known to function without any electrochemical surface preparation (Industry Advisory Group).

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 15 of 31

Therefore, the following approach was adopted:

1. Carry out preliminary X100 plating + adhesion trials as described in the initial proposal in order to demonstrate overall “plateability” and good adhesion to X100 steel
2. Proceed with the CA design process with the assumption that a tight mechanical fit of the CA to the pipe surface is sufficient to transfer load on crack impact; re-address in Phase II if required.


3.2.4.2 X100 Adhesion Results

Based upon Integran’s extensive experience with the surface preparation of metallic substrates for electroplating, a cleaning and activation protocol was selected and validated for X100 pipeline steel. This procedure consisted essentially of sand-blasting, alkaline cleaning, and acid dipping, with and without applied current. It has also been determined that the most practical means to evaluate coating adhesion is via simple bend testing followed by optical examination. Results of one such test of Nanometal-plated X100 can be seen in Figure 7 below.

It can be seen in Figure 7 that the Nanometal coating has fractured after 180° bending. This is to be expected given its high strength and moderate ductility. However, the key point to note is that, despite fracturing, the coating did not delaminate and disbond in any way. Rather, excellent adhesion was preserved throughout bending. This is a reliable indication that strong bonding to the X100 steel was achieved.



Figure 7 Photograph of a 1”-wide Nanometal-coated X100 steel strip bent by 180°. Optical examination revealed fracture of the coating material without delamination, indicative of high bond strength between the coating and substrate.

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 16 of 31

3.2.5 Task 2 Conclusions

In conclusion, Task 2 proof-of-concept Nanoplatting and mechanical testing of X100 pipeline steel yielded the following information:

1. Integran's ultrahigh strength nanocrystalline Co-P and Ni-Fe alloys are approximately twice as strong as X100 steel, even under high strain rate deformation conditions;
2. X100 pipeline steel can be Nanoplated with a coating bond strength that allows the coated component to survive tensile deformation until the ductility limit of the ultrahigh strength coating material and without delaminating from the substrate;
3. Effective load transfer (manifested as strengthening of the coated samples) can be successfully achieved between the Nanometal and the X100 steel. Whether an electrochemical activation process to X100 is required to create effective load transfer during the crack arrest event is not known, but it is the opinion of the Industry Advisory Group and CSM that an intimate mechanical bond (such as the one used for the incumbent composite-based CA designs) will likely be sufficient; and
4. Overall, Nanometal applied to X100 steel pipe has the ability to offer a marked structural reinforcement contribution to the pipe structure.

3.3 Task 3 – Finite Element (FE) Simulations

3.3.1 Introduction


Due to the exceptionally high cost (>\$0.5-2 million per test) of full-scale pipeline CA testing, considerable effort has been put into the accurate numerical simulation of the crack arrest event. While the running fracture scenario is extremely complex, a few organizations have succeeded in developing codes that are deemed to be reasonably reliable e.g. Engineering Mechanics Corporation (emc²), Battelle Labs, Advantica Ltd, and Centro Sviluppo Materiali (CSM). Through our Task 1 conversations with the Industry Advisory Group, it became clear that CSM, in particular, is in the forefront of this field and so a sub-contract was awarded to CSM for the purpose of evaluating Integran's nanometals as crack arrestor materials via FE modeling.

Task 3 was carried out as follows:

1. Preliminary calculations were performed at Integran to pre-screen for the subset of nanometal candidate alloys to be evaluated by CSM
2. Mechanical property data for these candidate Nanometal alloys was submitted to CSM
3. CSM then performed a set of 12 full Nanometal crack arrest simulations

3.3.2 Preliminary Calculations

As described in the Task 1 results, the overall technical, economic and practical installation feasibility of the monolithic Nanometal-based CA design hinges on the thickness of Nanometal required to stop the running fracture. In order to obtain a rough estimate for this figure, preliminary calculations were performed according to the method outlined in a recent paper on the optimization of mechanical crack arrestor design⁴. Mechanical property data from three Integran materials were employed in the calculations and the results as a function of Nanometal tensile strength can be found in Figure 8 below.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 17 of 31

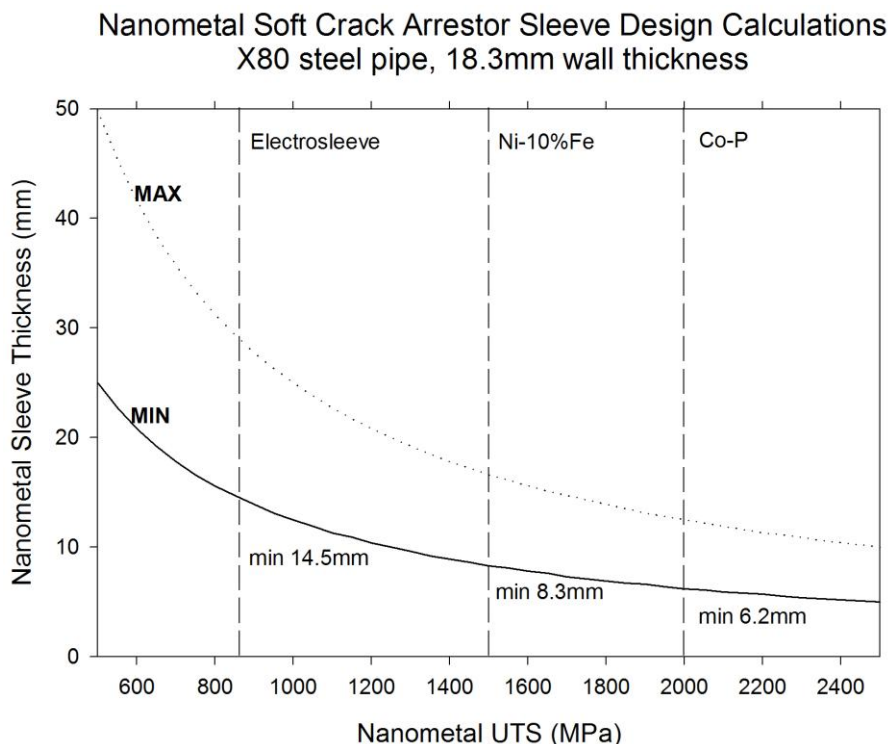



Figure 8 Preliminary calculations to evaluate the thickness of Nanometal, as a function of Nanometal tensile strength, required to arrest a running fracture in a “soft” manner (MIN curve) along with the maximum thickness beyond which “hard” arrest or “ring-off” would be likely to occur (MAX curve). The region between MIN and MAX is the region of interest for the design of “soft” CA’s in particular. 3 typical Nanometal alloys of varying strength were evaluated and typical X80 pipeline steel was used as the pipe material.

From Figure 8, it can be seen that less material is required to arrest a pipeline running fracture when an ultrahigh strength material (Co-P) is employed as compared to a weaker material (e.g. the standard Integran pipe repair material - “Electrosleeve”). This reinforces the importance of maximum CA material strength. Secondly, these preliminary calculations suggest that >6 mm thickness of Integran’s strongest material (Co-P) would be required to arrest the crack in a “soft” manner while a thickness greater than approximately 12 mm runs the risk of creating a “ring-off” event. This information, along with the monolithic Nanometal mechanical property data for all three materials, was conveyed to CSM as background for their detailed simulations.

3.3.3 Introduction to CSM’s PICPRO® Code


This introduction to the CSM pipeline crack arrest simulation has been taken from their final report, attached here as Appendix A.

“Ductile fracture propagation in gas pipelines has been widely studied in the last 30 years by several institutes, including Centro Sviluppo Materiali S.p.A. (CSM). In recent years, CSM’s capability in this area has been further enhanced through the development of a proprietary finite element code named

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 18 of 31

PICPRO® (PIpe Crack PROpagation) able to model ductile fracture propagation in buried or unburied gas pipelines^{14,15}. The code is able to take into account both steady-state and transient fracture propagation conditions as well as abrupt changes of constraint characteristics. It also considers local strain rate effects¹⁵, soil constraint effects¹⁶ and decompression of the gas flowing out from the fracture breach according to the actual gas composition, pressure and temperature. Moreover, the implementation of an additional tool allows PICPRO® to account for the presence of Crack Arrestors (CA) along the pipeline, thus estimating the resulting effect of the device on the running shear fracture. Different types of CAs can be modeled and simulated, including clamps, rings, ropes, steel sleeves with or without grout (epoxy resin or concrete), thicker wall pipes and composite sleeves. PICPRO® has been successfully used to perform numerical predictions of the results of recent experimental burst tests performed on X100 and X120 large diameter pipelines, such as the BP test¹⁷, the Demopipe 2nd test¹⁸ and the URC¹⁹ test, the latter being the only full-scale burst test conducted on X120 pipes (conducted by CSM on behalf of URC). The agreement between numerical predictions and experimental results were excellent, thus demonstrating the capability of the CSM code to correctly simulate the ductile fracture event and the CA effectiveness.”

For each simulation, the influence of the CA on the running fracture is captured schematically in a figure such as the one in Figure 9 below. Crack propagation runs from the left-hand side of each figure and through to the right-hand side. See Appendix A for a complete explanation.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 19 of 31

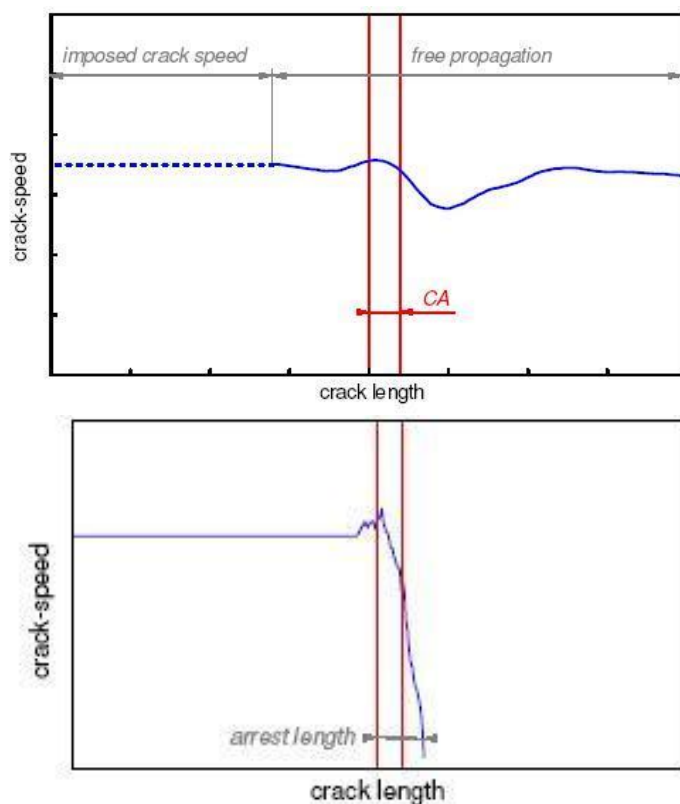



Figure 9 Two typical PICPRO® outputs. The incoming crack speed is found on the left hand side of the figure. (a) the CA did not arrest the crack (failure to arrest); and (b) the CA did not arrest the crack within the CA, but slowed it down such that it was unable to propagate further upon exiting the CA (successful arrest).

3.3.4 Introduction to CSM's Nanometal CA Simulations

A total of 12 PICPRO® simulations were performed involving 2 levels of monolithic Nanometal CA wall thickness, 2 levels of CA axial length and 3 levels of crack speed. These experimental details are outlined in Table 1 below. The term “Levels” refers to the total number of experimental settings for the independent variable in question. For example, CA performance was evaluated at low, medium and high crack speeds, for a total of 3 “Levels”. The majority of the simulations were performed using nanocrystalline Co-P as the CA material, but some direct comparisons were made to Integrant’s Ni-Fe alloy, which yielded similar crack arrest performance in the majority of cases. See Appendix A for more detail.

It should also be noted that dialogue with CSM revealed that the original presumption that strong bonding of the Nanometal to the steel pipe substrate might not, in fact, be as critical as once thought. In other words, it has been the experience of CSM that as long as the CA is applied close to the pipe surface, the required load transfer to the CA will occur as desired. Hence, this “bond strength” feature of the

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 20 of 31

Nanometal-based design was not considered in the FE simulations, and it remains to be seen whether this feature can be used to improve the effectiveness of the CA or not.

Table 1 Experimental design for Nanometal CA simulations

Levels of CA Length	Levels of CA Wall Thickness	Levels of Crack Speed	Total Number of Simulations
2 (500 – 1000 mm)	2 (3 – 6 mm)	3 (200 – 275 – 300 m/s)	12

The sleeve arrestors were considered as mounted along an ISO 3183 / API 5L²⁰ X100 grade steel pipeline, 36” outer diameter, 20.0mm wall thickness, operated at 226bar with natural lean gas (predominately methane). Pressure value corresponds to a design factor of 0.75 of SMYS (Specified Minimum Yield Strength).

3.3.5 Results of CSM Finite Element Simulations

The FE survey consisted of 12 simulations involving monolithic Nanometal CA’s of 2 different wall thicknesses and axial lengths (see Table 1). In other words, four different CA geometries were investigated:

1. CA of 500mm length and 3mm thickness (1 layer)
2. CA of 1000mm length and 3mm thickness (1 layer)
3. CA of 500mm length and 6mm thickness (2 layers)
4. CA of 1000mm length and 6mm thickness (2 layers)

The performance of each device was then investigated by simulating 3 different crack speeds representative of a wide range of actual in-field running fractures: 200, 275 and 350m/s.

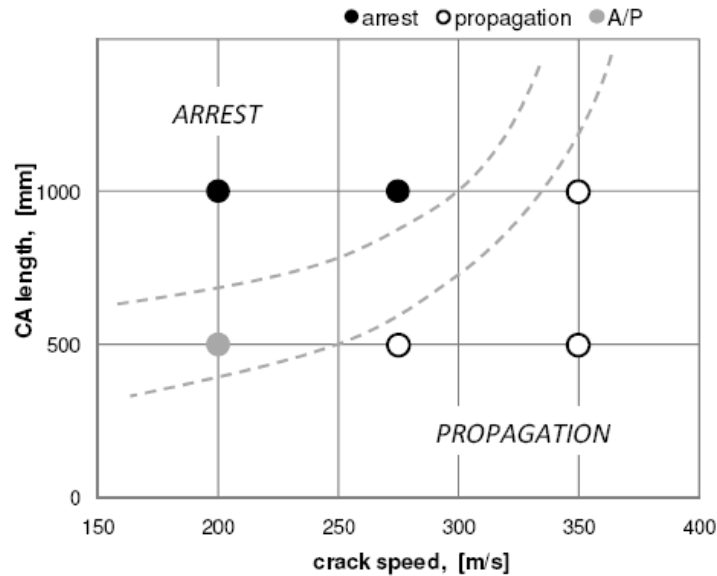
The reader is directed to Section 5.1 of the full CSM report in Appendix A for PICPRO® outputs similar to those shown in Figure 9 and corresponding to each of these CA designs. In the interest of brevity, all the simulation results have been collated in Figure 10 below (Section 5.5 of the CSM report).

The following conclusions were drawn from the FE results summarized in Figure 10:

1. The 3mm-thick CA with an axial length of 0.5m is ineffective in arresting the propagating crack;
2. Simply increasing the axial length of this 3mm-thick design up to 1m does not appear to be an effective remedy since it only stops cracks moving at incoming speeds lower than 200m/s;
3. On the other hand, the increase of the CA thickness to 6mm produces significant improvements (see Figure 10(b)). The device appears to be able to lead the fracture to a rapid arrest, although some uncertainties remain for the half-meter long arrestor if high speeds are accounted.

Hence, the overall conclusion is that approximately 6mm of Integran’s highest strength Nanometal (either Co-P or Ni-Fe as both have similar mechanical properties, see Figure 5(a)) appears to be intrinsically capable of arresting running fractures in high strength X100 pipeline steel.

SINGLE LAYER ARRESTOR – INDICATION FOR DESIGN



2-LAYERS ARRESTOR – INDICATION FOR DESIGN

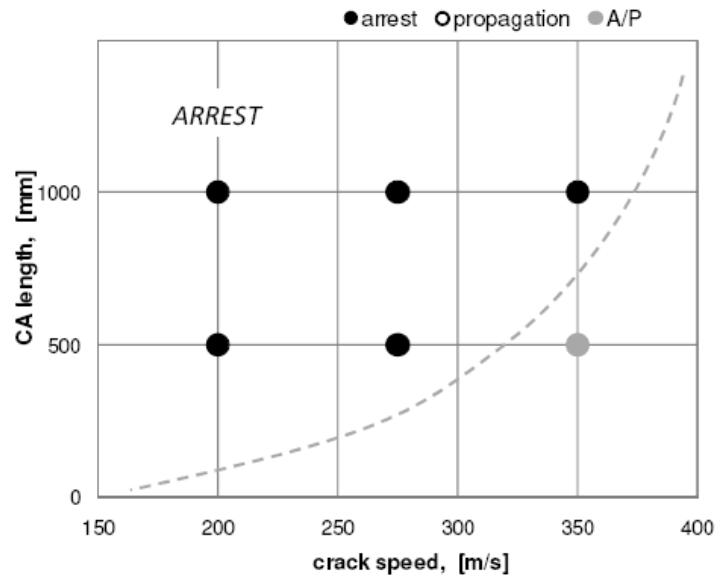



Figure 10 Overall summary of the simulation results for both the (a) 3mm-thick and (b) 6mm-thick Nanometal CA designs. “Single layer” = 3mm thickness. “2-Layers” = 6mm thickness.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 22 of 31

3.3.6 Some Comments on “Soft” vs. “Hard” Arrest

As discussed, the FE simulations carried out by CSM were quite useful in demonstrating those conditions under which Nanometal-based CA’s can stop running fractures. However, they do not fully address the specific objective of this program, namely the design of a CA created for the specific purpose of not only arresting fractures, but arresting them in a “soft” manner. Discussions with CSM were conducted on this topic and an excellent summary of the situation was provided by Andrea Meleddu of CSM, an excerpt of which can be found below:

“In order to achieve a *soft* crack slow down, the arrestor thickness cannot be too thick or else it will impart an excessive constraint action. On the other hand, the objective of arresting the crack within the device length necessitates adequate design thickness in order to ensure sufficient mechanical constraint against the crack opening.

For this purpose, please consider the picture below [Figure 11]. The upper plot considers 2 CA’s of identical thickness but different lengths. Roughly speaking, the initial slow down slope is similar for both, but the longer is the device, the longer is the time at which such slow down takes place. That’s why the 1.0m long CA leads to the arrest, while continued propagation is observed for the 0.5m CA. In the lower plot, 2 CA’s of identical length but different thickness are compared. In this case, the thicker the arrestor, the more severe is the crack slow-down.

Therefore, roughly speaking, the arrest length is a function of the device length (upper plot), while the slope of the crack slow-down or deceleration is a function of the CA wall thickness (lower plot).

In this sense, PICPRO® is able to properly provide indications about the rate of crack deceleration, thus demarcating those instances of abrupt arrest (which are more prone to ring-off occurrence) and those of long arrests, which may be referred to as “soft”. In this light, it may be the case that some of the “uncertainty cases” [grey dots in Figure 10] can be considered to be as soft arrest, provided the arrest occurs within the CA.

However, it is important to note that the determination of a criteria for the definition of the “dangerous” and the “safe” region is a matter of great concern, since ring-off is a very dynamic phenomenon originating from the concomitance of various events, not all being of easy interpretation. Hence, arguing the grade of exposure to “ring-off”, on the basis of the sole speed slow-down tendency is not advisable. In other words, *a more reliable “soft arrest” predictive capability can only be gained through dedicated study of this topic, including literary review, deep investigation and experimental analysis.*”

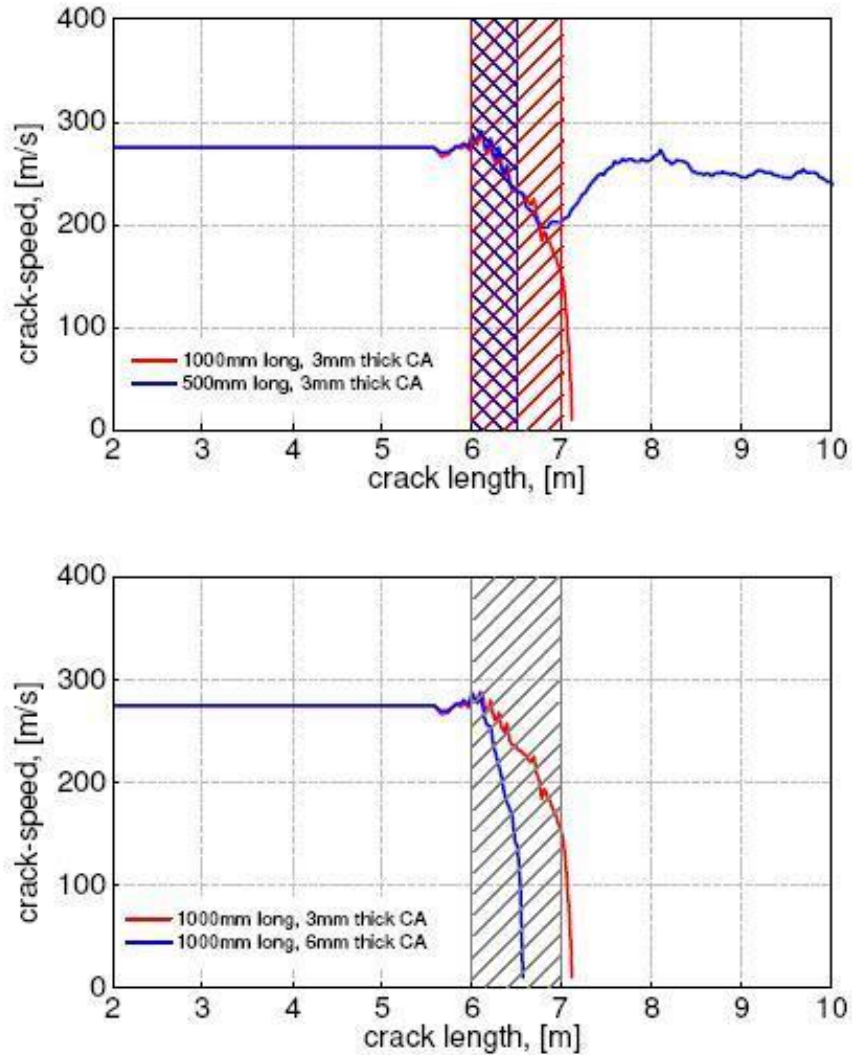



Figure 11 Schematic of how CSM's simulation technique could potentially be used to shed light on the nature of "soft" vs. "hard" arrest. Roughly speaking, the arrest length is a function of the device length (upper plot), while the slope of the crack slow-down or deceleration is a function of the CA wall thickness (lower plot).

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 24 of 31

3.3.7 Task 3 Conclusions

1. The FE survey has proved to be an extremely useful tool insofar as it has provided important information concerning the general effectiveness and ability of nanostructured metal alloys to arrest running fractures in high strength pipeline steel. In particular, ultrahigh strength Nanometal alloys appear to be intrinsically strong and tough enough to be employed as pipeline CA materials.
2. The FE survey yielded meaningful information concerning the influence of CA geometry on its ability to oppose shear fracture propagation. The most important geometrical design feature of the CA is its thickness, and approximately 6mm of Nanometal would be required to arrest a propagating fracture.
3. CA length is also an important geometrical design feature, but since both settings evaluated (0.5m and 1m) are virtually equivalent from a practical and economic feasibility perspective, the broader concept feasibility does not hinge primarily upon this design variable.
4. The PICPRO® code represents an extremely useful tool that, if possible, should be used to help guide the design of the Nanometal-based CA.
5. In its present form, the code is not capable of assessing the performance of CA's that are of a non-uniform thickness profile (such as the "bell-shaped" Nanometal CA profile mentioned earlier), nor can it accommodate multimaterial CA's. However, CSM has expressed willingness to modify their code to accurately predict the performance of these more sophisticated CA concepts as part of the Phase II effort.

3.4 Overall Phase I Conclusions


The Phase I summary has been broken down into the primary technical, practical in-field installation, and economic feasibility conclusions outlined below:

3.4.1 Technical Feasibility

From the work performed in the Phase I portion of this program, it can be concluded that Integran's electrodeposited Nanometal appears to be intrinsically strong and tough enough to effectively retard running fracture propagation in high strength pipeline steel. The requisite bond strength of the Nanometal CA to the steel pipe is yet to be determined, but dialogue with experts has indicated that there may be no need to apply an electrochemical cleaning process and that a tight mechanical fit (e.g. tight wrapping as is done for composite-based CA's) is likely sufficient to effectively transfer the crack opening load.

3.4.2 Practical In-Field CA Installation Feasibility

It is unlikely that the originally proposed concept of *in situ* electroforming a monolithic layer of high strength Nanometal onto the pipe surface will be practical for in-field use because 6mm+ thickness requires >20hrs to electrodeposit. However, it is proposed that a bell-shaped (in cross-section) design whereby Nanometal foil is wrapped around the pipe OD until the central portion meets or exceeds the equivalent strength of the monolithic 6mm CA could be practical for in-field installation. This presumption is based largely on the knowledge that the ClockSpring™ design, which is similar in its installation, is a commonly used CA solution. Figure 12 below contains a photograph of one of

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 25 of 31

Integran’s continuous foil plating tools, along with a photograph of the ultrahigh strength Ni-Fe foil that is routinely produced on it.

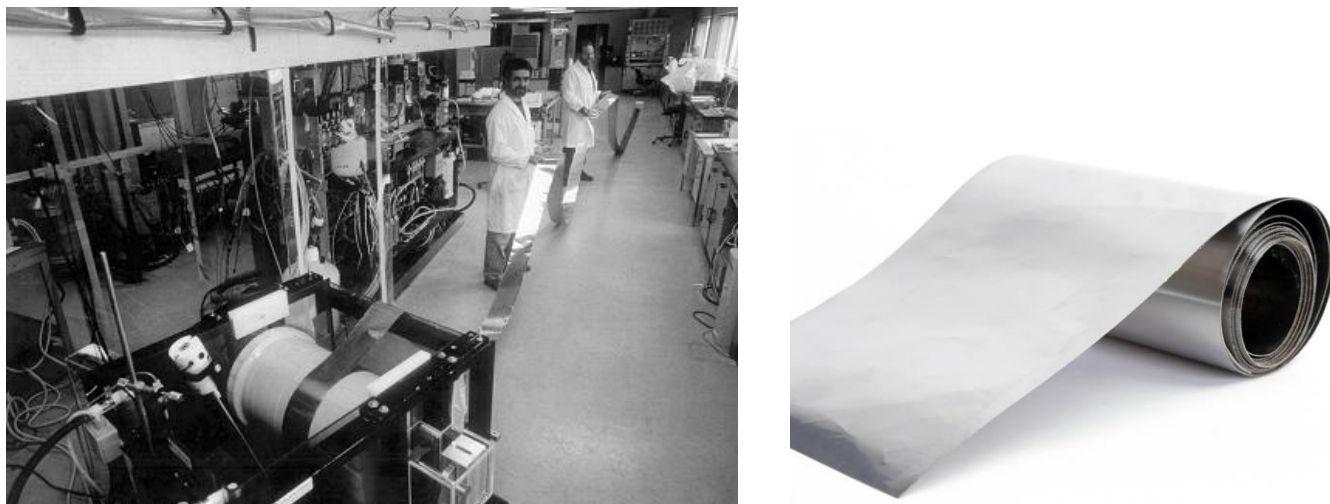



Figure 12 Integran’s continuous Nanometal foil production process.

3.4.3 Economic Feasibility

The benchmark cost figure provided by the Industry Advisory Group was, roughly speaking, \$10-20k per CA. It is estimated that the total installation cost for the Nanometal-based CA will be 10x raw metal cost. It should be emphasized that this is only a ballpark figure. A 0.7m-long “wrapped foil” nanocrystalline Ni-20Fe CA built up to a thickness equivalent to 6mm of monolithic material would weigh less than 100kg, which would cost approximately \$2640 in raw metal costs (assuming Ni costs \$15/lb). Thus, the ballpark total cost of the Nanometal-enabled soft crack arrestor is, very roughly speaking, \$26,000 each. Fortunately, fabrication of ultrahigh-strength Nanometal foil is a well-established process at Integran (see Figure 12) and so it is very likely the case that the end-user cost targets can be met even if they fall below \$26,000. For the purposes of this Phase I effort, however, it can be concluded that the cost of the Nanometal CA should fall at approximately \$20k, with potential for refinement of this figure. The value associated with the “soft” arrest characteristic of the CA design is not known. In other words, while \$10-20k is the ballpark price point for conventional CA’s, the value associated with the specific performance enhancement of significantly improved frequency of “soft” arrest as opposed to “ring off” has not yet been established.

 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 26 of 31

4.0 PHASE II ACTIVITIES

Based upon what has been learned in Phase I, it is our view that the processing flexibility inherent to electroforming should permit the CA designer to grade the Nanometal thickness in a bell shape (viewed in cross-section, see Figure 13 below) and that, with such a geometry, the crack can be arrested within the CA and not at the CA edge. This simple geometrical feature should significantly improve the probability of achieving a “soft” arrest as opposed to “ring-off”. Unfortunately, this is merely a presumption and would need to be proven as follows:

4.1 Task 1 – Development of Nanometal-Enabled CA Design

Since the originally proposed *in situ* monolithic Nanometal CA design is impractical, it is proposed that equivalent strength and the desired non-uniform “bell-shaped” cross-sectional CA profile can be achieved by tightly wrapping ultrahigh strength Nanometal foil around the pipe OD in a fashion similar to the ClockSpring™ technology – see Figure 13 below for an illustration of this concept. It is anticipated that, in addition to the foil geometry with respect to thickness and cross-sectional area across the CA length, one of the most important design parameters in such a design would be the resin used to bind the Nanometal multilayers. Preliminary discussions with colleagues at Carnegie-Mellon University have suggested that a high energy-absorption UV-curable polyurethane currently under development might be suitable for such a purpose. Alternatively, it may be the case that standard fiberglass resins are sufficient. Regardless, it seems likely that a vacuum molding process such as that used to construct fiberglass boat hulls would represent the best way to eliminate all voids from the multimaterial CA structure.

Owing to the extreme component shape flexibility inherent to electrodeposition, other Nanometal-enabled CA designs have also been envisioned. Similar to the “wrapped foil” concept described above, another possible design involves the fabrication of fiber-reinforced composite structures (again, quite similar to the ClockSpring™ technology) where the strength and toughness of the CA have been significantly enhanced by the utilization of Nanometal-coated ultrahigh strength fibers. These high tenacity fibers would then be bound or woven together in a fashion similar to standard fiber-reinforced composite materials such as fiberglass. In other words, the proposed design consists of Nanometal-coated ultrahigh strength / toughness fibers as the load bearing members in a fiberglass-like composite structure. The advantage of this concept vs. incumbent composite solutions would be significantly enhanced strength and toughness, owing to the metallic character of the fibers. A schematic of this concept is illustrated in Figure 14 below along with a photograph of an existing ultrahigh strength fiber production line currently running at Integran. Similar to the foil production process, Nanometal-coated fibers have been an area on on-going development at Integran and so it may be the case that this experience could be leveraged to produce a design that is technically, practically, and economically palatable to end users.

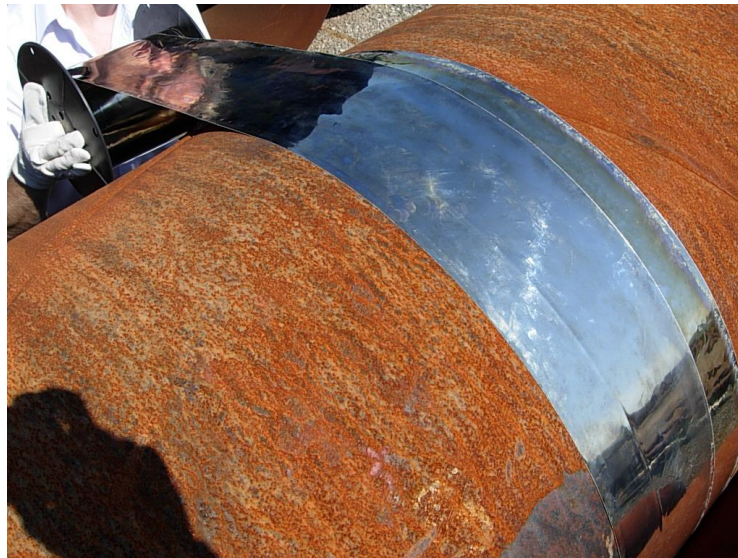


Figure 13 (top) Photograph of the Nanometal “wrapped foil” concept; and (bottom) schematic illustrating the “bell-shaped” non-uniform thickness profile that is proposed could yield improved “soft” arrest performance.

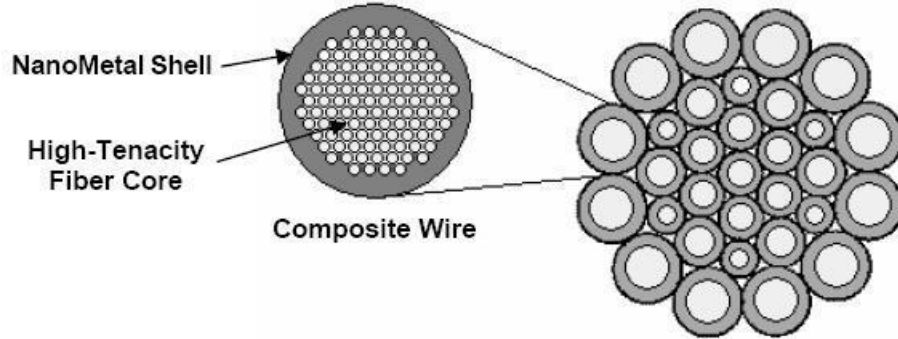



Figure 14 (Top) schematic of the Nanometal-enabled high tenacity fiber design that could potentially be used to impart both strength and toughness to a novel fiber-reinforced composite CA design; and (bottom) photograph of the ultrahigh strength Nanometal-coated fiber reel-to-reel production line currently running at Integrin (fiber travel direction is left to right).


 TECHNOLOGIES U.S.A. INC.	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 29 of 31

4.2 Task 2 - Modification of CSM Code to Guide CA Design

As mentioned earlier, the PICPRO® code cannot account for the effect of non-uniform CA thickness on the CA performance. In addition, if the “wrapped Nanometal foil” or “high tenacity fiber”-based concepts described above prove promising, the code will also not be able to simulate these designs. However, CSM is willing to improve and modify their code and this would be built into the Phase II proposal.


4.3 Task 3 - Larger Scale Burst Testing

In addition to FE simulations, CSM has extensive experience with scaled-up burst testing of prototype CA concepts. Hence, Phase II would see the Nanometal-based design burst tested in a sub-scale pipe. CSM could potentially take part in this activity as well, or a 3rd party could also be engaged for this purpose.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 30 of 31

5.0 REFERENCES

- ¹ Brauer, H., G. Knauf, and H.-G. Hillenbrand. 2004. Crack arrestors. *In* Proc. 4th Int. Conf. On Pipeline Technology, 9-12 May 2004, Ostend, Belgium.
- ² Fonzo, A., A. Meleddu, M. Di Biagio, G. Mannucci, G. Demofonti, C.W. Petersen, and N.E. Biery. 2006. Crack propagation modeling and crack arrestor design for X120. *In* Proc. 2006 International Pipeline Conference, September 25-29, 2006, Calgary, AB, Canada (American Society of Mechanical Engineers, New York, NY), paper IPC2006-10319.
- ³ Takeuchi, I, H. Makino, S. Okaguchi, N. Takahashi, and A. Yamamoto. 2006. Crack arrestability of high-pressure gas pipelines by X100 or X120. *In* Proc. 23rd World Gas Conference, Amsterdam.
- ⁴ Wilkowski, G., D. Rudland, and B. Rothwell. 2006. How to optimize the design of mechanical crack arrestors. *In* Proc. 2006 International Pipeline Conference, September 25-29, 2006, Calgary, AB, Canada (American Society of Mechanical Engineers, New York, NY), paper IPC2006-10357.
- ⁵ G. McMahon and U. Erb, *Microstr. Sci.*, **17** (1989) 447
- ⁶ G. McMahon and U. Erb, *J. Mat. Sci. Lett.*, **8** (1989) 865
- ⁷ F. Gonzalez, A.M. Brennenstuhl, G. Palumbo, U.Erb and P.C.Lichtenberger, *Mat. Sci. Forum*, 225-227 (1996) p.831
- ⁸ G.Palumbo, F.Gonzalez, A.M.Brennenstuhl, U.Erb, W.Shmayda and P.C.Lichtenberger, *Nanostructured Materials*, 9 (1997) p. 737
- ⁹ G.Palumbo, P.C.Lichtenberger, F.Gonzalez and A.M.Brennenstuhl, U.S. patents 5,527,445 (1996); 5,516,415 (1996); 5,538,615 (1996)
- ¹⁰ U. Erb and A. M. El-Sherik, US Patent No. 5,352, 266 (1994)
- ¹¹ U. Erb, A.M. El-Sherik, C. Cheung, M.J. Aus, US Patent No. 5,433,797 (1995)
- ¹² U. Erb, K.T. Aust, & G. Palumbo, in *Nanostructured Materials: Processing, Properties, & Applications*, 2nd edition, C.C. Koch (ed.), William Andrew, Inc., Norwich, NY, 2007; p. 235
- ¹³ C.C. Koch, *J. Metast. Nanostr. Mat.*, **18**, (2003), 9
- ¹⁴ G. Berardo, P. Salvini, G. Mannucci, G. Demofonti, "On Longitudinal Propagation of a Ductile Fracture in a Gas Line Pipe: Numerical and Experimental Analysis" Intern. Pipeline Conference IPC2000, Calgary, October 2000.
- ¹⁵ A. Fonzo, P. Salvini, M. Di Biagio, G. Mannucci, "Full history burst test through finite element analysis" , Proc. of the 4th Inter. Pipeline Conf., Calgary 2002, paper n. IPC2002-27120.
- ¹⁶ P. Salvini, G. Berardo, G. Demofonti, G. Mannucci, "Numerical model of the backfill constraint effect during fracture propagation on buried large diameter gas pipelines", Pipeline Technology Conference 2000, Brugge 22-24 May 2000.
- ¹⁷ G. Mannucci, M. Di Biagio, G. Demofonti A. Fonzo, P. Salvini, A. Edwards, "Crack arrestor design by finite element analysis for X100 gas transportation pipeline", Proceedings of the 4th International Conference on Pipeline Technology, 9-13 May 2004, Ostend, Belgium.
- ¹⁸ G. Demofonti et al., 2004, "Fracture propagation resistance evaluation of X100 TMCP steel pipes for high pressure gas transportation pipelines using full-scale burst tests", Proc. of the 4th International Conference on Pipeline Technology, Ostend, Belgium.
- ¹⁹ A. Fonzo, A. Meleddu, M. Di Biagio, G. Mannucci, G. Demofonti, C.W. Petersen, E.B. Biery, "Crack propagation modeling and crack arrestor design for X120", Proceedings of the 6th International Pipeline Conference, September 25-29, 2006, Calgary, Alberta, Canada.
- ²⁰ ISO 3183:2007 (E) "Petroleum and natural gas industries – Steel pipe for pipeline transportation systems", 2007.

	Crack Arrest and Structural Repair of High Strength Steel Piping by In-Situ Sleeving with Nanostructured Materials SBIR Phase I Final Report		
Contract No: DTRT57-08-C-10022	Project No: 1A-7029	Date of Issue: 2008-07-31	Page 31 of 31

**6.0 APPENDIX A – FINAL REPORT FROM CENTRO SVILUPPO MATERIALI S.P.A.:
“ADOPTION OF NANOSTRUCTURED METAL ALLOY IN CRACK ARRESTOR
DESIGN – FEASIBILITY STUDY BY FEM ANALYSIS”**

CSM Ref. No. S C140 005
CSM Report No. 13685R

Research for
Integran Technologies USA

Adoption of nanostructured metal alloy in crack arrestor design

Feasibility study by FEM analysis

FINAL REPORT

A. Meleddu

A. Fonzio

July 2008

All data or information referred to in the present report shall be considered strictly confidential. Any use or disclosure, even if partial, shall be subject to prior agreement between Contractors and CSM.

CONTENTS

1	BACKGROUND	1
2	INTRODUCTION	2
3	FUNDAMENTALS OF CA NUMERICAL SIMULATIONS	3
	3.1 Procedure for CA simulation	3
	3.2 Representation of CA design criteria	5
4	CA GEOMETRIES.....	7
5	RESULTS OF PICPRO® SIMULATIONS	9
	5.1 3mm thick, 500mm long CA.....	11
	5.2 3mm thick, 1000mm long CA.....	12
	5.3 6mm thick, 500mm long CA.....	13
	5.4 6mm thick, 1000mm long CA.....	14
	5.5 Resume of results	15
	5.6 CoP to NiFe metal alloy comparison	16
6	DISCUSSION AND CONCLUSION	18
	REFERENCES.....	20

1 Background

Ductile fracture propagation in gas pipelines has been widely studied in the last 30 years by several institutes, including Centro Sviluppo Materiali S.p.A. (CSM). In recent years, CSM's capability in this area has been further enhanced through the development of a proprietary finite element code named PICPRO[®] (PIpe Crack PROpagation) able to model ductile fracture propagation in buried or unburied gas pipelines [1,2].

The code is able to take into account both steady-state and transient fracture propagation conditions as well as abrupt changes of constraint characteristics. It also considers local strain rate effects [2], soil constraint effects [3] and decompression of the gas flowing out from the fracture breach according to the actual gas composition, pressure and temperature.

Moreover, the implementation of an additional tool allows PICPRO[®] to account for the presence of Crack Arrestors (CA) along the pipeline, thus estimating the resulting effect of the device on the running shear fracture. Different types of CAs can be modeled and simulated, including clamps, rings, ropes, steel sleeves with or without grout (epoxy resin or concrete), thicker wall pipes and composite sleeves.

PICPRO[®] has been successfully used to perform numerical predictions of the results of recent experimental burst tests performed on X100 and X120 large diameter pipelines, such as the BP test [4], the Demopipe 2nd test [5] and the URC [6] test, the latter being the only full-scale burst test conducted on X120 pipes (conducted by CSM on behalf of URC). The agreement between numerical predictions and experimental results were excellent, thus demonstrating the capability of the CSM code to correctly simulate the ductile fracture event and the CA effectiveness.

2 Introduction

In the ambit of the development of new devices for controlling the ductile fracture propagation on gas transportation pipelines, Integran Technologies is currently interested in evaluating the feasibility of adopting nanostructured metal alloys for constructing high performance Crack Arrestors (CAs) for arresting ductile fracture propagation in pressurized large diameter pipeline.

Thanks to their fine mechanical properties, such materials may supply the line with a considerable constraint action, thus increasing effectively the local fracture resistance.

According to this, Integran Technologies asked CSM to undertake a Finite Element analysis by using its own code PICPRO[®] with the aim of investigating the effectiveness of sleeve CAs made of nanostructured metal alloys in an adequate range of geometries.

A number of 12 simulations have been performed involving 2 levels of CA wall thickness, 2 levels of CA axial length and 3 levels of crack speed.

The sleeve arrestors are considered as mounted along a X100 grade steel pipeline, 36" outer diameter, 20.0mm wall thickness, operated at 226bar with natural lean gas (predominately methane). Pressure value corresponds to a design factor of 0.75 of SMYS (Specified Minimum Yield Strength).

The survey provided significant indications about the effectiveness of nanostructured metal alloys and gave meaningful details about the influence of CA geometry on its capability of opposing the shear fracture propagation.

Nevertheless, while the code capability in simulating conventional arrestors on large-diameters/high-grade pipelines is confirmed by experimental evidences, no test has been performed on nanometal reinforcements, which may support the code predictions whether nanometal reinforcements are considered. Experimental activity is thus recommended to verify and strengthen the PICPRO[®] predictive indications.

Within the present document CSM supplies Integran Technologies with the results of simulations, interpretation of the outcomes and indications about the effectiveness of the selected CA geometries in arresting a ductile fracture propagation.

3 Fundamentals of CA numerical simulations

PICPRO[®] has been developed to simulate the ductile fracture propagation upon a gas pressurized pipeline. Thanks to specific algorithms, the code takes into account both steady-state and transient crack propagation conditions and it considers the soil constraint effect, the gas decompression through the breach according to its actual chemical composition, pressure and temperature.

An additional tool is dedicated to the computation of the reciprocal interaction between the main pipe and an arrestor mounted along the line, thus providing predictions about the capability of the device in arresting a longitudinal ductile fracture. Since various arrestor typologies and geometries can be accounted, a correct use of the code furnishes useful indications about the optimal CA type and geometries (length, thickness, radial clearance, etc.).

The good predictive capability of PICPRO[®] has been successfully demonstrated by comparing the numerical predictions with actual full-scale test results [4,5,6].

In the following sections, the conceptual approach adopted for simulating the fracture propagation in presence of CA is presented (§3.1) as well as the representation of the CA design criteria (§3.2).

3.1 Procedure for CA simulation

For a given fracture propagation speed, PICPRO[®] is able to correctly model the stress-strain field acting at the pipe crack tip and to calculate any crack speed change due to the pipe material properties and/or the constraint action exerted by the crack stopper.

The hypotheses adopted for simulating the CA action are the following:

- arrestor rupture occurs when its material tensile strength is achieved. No Fracture Mechanics model is implemented into the CA model;
- no friction is considered between CA and pipe surface;
- flexional stiffness in longitudinal and hoop direction of the CA is neglected;
- running crack only propagates along longitudinal direction. No effects due to circumferential fracture deviation or encircling potentially causing a severance are considered.

The code simulates dynamic ductile fracture propagation in a buried gas pipeline on the basis of the balance between two opposing actions:

- the fracture *driving force*, representing the phenomena promoting the crack advance. It essentially consists of the gas flow through the breach and it is calculated by the code itself;
- the *resistance force* opposed to the fracture. It mainly consists of the inherent material resistance and the constraint action exercised by the soil around the line. The first is represented by a critical parameter characterizing the material resistance to shear fracture and it is measured through laboratory tests according to specific procedures [7]. Among those parameters, one of the most promising is the Crack Tip Opening Angle (CTOA), which indicates the edge opening in correspondence of the crack tip [8].

Whether the material toughness is unknown, PICPRO[®] deserves an alternative approach, which is particularly appropriate for those classes of pipe steels for which laboratory-to-pipe toughness transferability models are lacking or are not yet fully reliable, as the high grade steels (\geq API X80).

It is based on the fact that, once that pipeline geometry, gas composition and operative conditions are given, crack steady state propagation speed only depends on pipe material toughness or, that is the same, material toughness is strictly connected to the steady state propagation speed. This allows to consider the “*crack speed*” equivalent to the unknown “*inherent fracture propagation material resistance*”, and to adopt it for the crack arrestor design.

This is the approach adopted in both past [4, 6] and present works and it is schematically described in Figure 1.

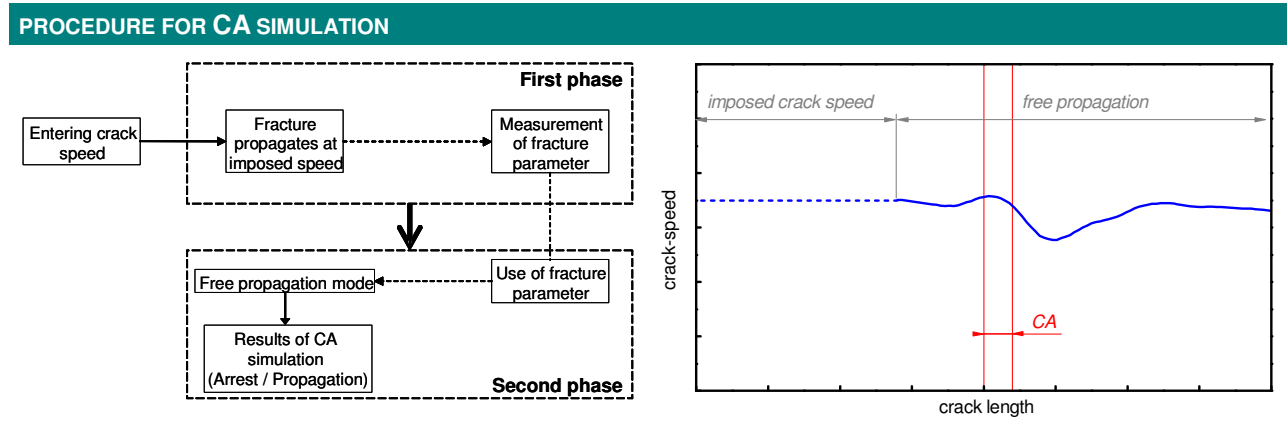


Figure 1 - Finite Element simulation procedure by means of PICPRO®

Figure 2 – example of PICPRO® result (no arrest predicted for the example in figure).

First, a constant crack speed is imposed until the stability of all phenomena is achieved. Under these conditions of steadiness the fracture “*driving force*” exactly equals the pipeline “*resistance force*”. In other words the *actual* value of the fracture parameter (such as the Crack Tip Opening Angle) is equal to its *critical* value. This value is measured and stored by the code. It corresponds to the *inherent fracture propagation material resistance*.

Afterwards, the *free propagation mode* is imposed, which allows the crack to propagate freely. Crack speed is not more imposed but calculated on the basis of the balance between the energy applied to the fracture (*driving force*) and the energy resistance value (previously measured). Nevertheless, since no local constraint changes occurred, crack continues to run at the same speed as previously until it reaches the CA edge.

As fracture enters the arrestor, deceleration/arrest or further propagation within the device are predicted according to the influence of the CA on the crack speed. If the constraint effect is sufficient, deceleration or arrest occurs; if not, a further propagation behind the CA is predicted.

PICPRO® estimates the crack speed evolution before, along and behind the CA as well as its status, thus predicting whether it suffers any damage and the length of the broken region.

An example of result is shown in Figure 2 where the two following phases of “*imposed crack-speed*” and “*free propagation*” are indicated. In this particular case, the constraint action of the CA, though determining a fracture slow-down, is not enough to lead to an arrest and the crack propagates beyond the device.

3.2 Representation of CA design criteria

Simulations are performed by imposing various values of initial crack speed in an appropriate range as to cover the typical speed of shear fracture propagation on a pressurized pipeline (about 80-350m/s).

For each crack speed, the effectiveness of a given CA typology is investigated by varying the geometrical parameter to be designed (wall thickness, axial length, etc.) in the field of interest.

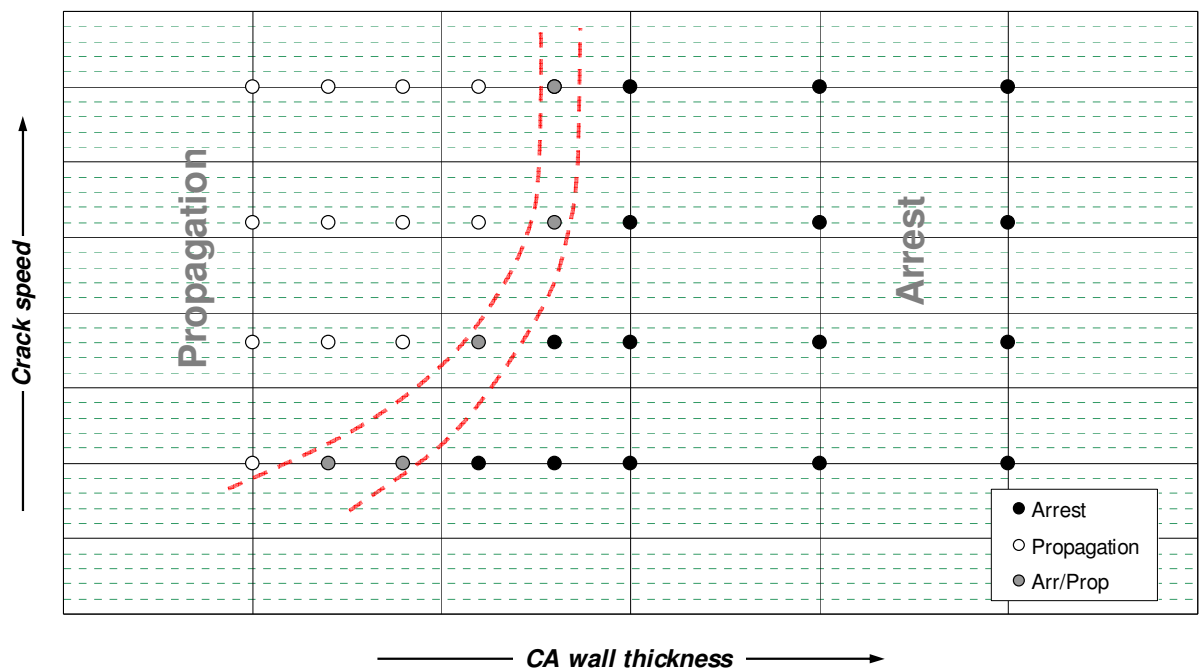
The results are summarized in a graph where fracture *arrests* and *propagations* predictions are depicted with different colors. In Figure 3 an example is reported where the CA wall thickness is the design parameter.

- White dots correspond to an insufficient effectiveness of crack arrestor in stopping the propagating fracture (for the wall thickness/fracture-speed specific condition).
- Black dots stand for successful results.
- Some outcome may also happen, which cannot be regarded neither as clear *arrest* nor a *propagation*. They mainly consist of those occurrences where the crack stops behind the CA or whether a very low propagation speed is predicted to occur. In first case, though an arrest is predicted shortly behind the device, the crack escape from the arrestor may be considered an undesirable event. In the second, it has to be pointed out that crack propagating with speeds lower than about 80m/s are hardly probable, since at such low speeds, instability phenomena have high probability to occur, which may induce fracture redirection, spiralization and consequent arrest. For those reasons, such results are regarded as *uncertain cases* and are represented with a grey dot in the plot.

By means of this diagram the conditions of “fracture arrest” and “fracture propagation” are split in two regions so that an indication of the minimum required CA wall thickness as function of the fracture entering speed is given.

It has to be noted that similar diagrams can be realized by considering two different geometrical parameters (one on each axis) under the same crack propagation speed.

Further information about all the simulation results can be furnished in a detailed table where all the specific kinematical values are provided such as: fracture outgoing crack speed (if any), CA damaged length, etc.



4 CA geometries

In agreement with Integran Technologies, a study has been performed aimed to provide useful indications about the suitability of using nanocrystalline metal alloys for constructing crack arrestors. The devices consist of a number of layers wrapped around the main pipe as to provide an additional constraint action to the main pipe body. Various geometries have been considered differing each other in the axial length and the number of layers (that is Crack Arrestor wall thickness).

In details, 2 values of axial length and 2 values of wall thickness have been investigated. The arrestors is considered to be applied on a ISO 3183 / API 5L [9] X100 grade steel pipeline with the geometry reported in Table 1.

PIPE OD	PIPE WALL THICKNESS	STEEL GRADE	OPERATING PRESSURE	DESIGN FACTOR	GAS
[in]	[mm]	API 5L grade	[bar]		
36	20.0	X100	226	75%	natural

Table 1 – pipeline geometry

Moreover, since the CA capability in arresting a ductile fracture propagating along a gas pipeline is strictly connected with the speed of the fracture itself, 3 levels of crack-speed have been considered in order to evaluate the device attitude along a range of severe conditions.

The number of simulations are summarized in the following Table 2.

LEVELS OF CA AXIAL LENGTH	LEVELS OF CA WALL THICKNESS	LEVELS OF CRACK SPEED	TOTAL NUMBER OF SIMULATIONS
2 (500-1000mm)	2 (3-6mm)	3 (200-275-350m/s)	12

Table 2 – agreed layout for FEM simulations

Concerning the material to be considered for the CA, Integran Technologies proposed to CSM three different materials: a pure Ni, a NiFe alloy and a CoP alloy. Their mechanical properties are reported in the Table 3 as provided by Integran.

On the basis of the provided data the cobalt-based nanometal has been chosen, since it has been considered as the most performing among the proposed alloys. In fact, the very high tensile properties (in terms of yield and tensile strength) and considerable ductility (see the elongation to failure), are of most relevance for the material ability in supporting an effective crack slowing-down and arrest.

However, since NiFe alloy owns interesting mechanical properties as well, the effectiveness of the two materials have been roughly compared upon a limited number of simulations (see §5.6).

PROPERTY	nNi-ES-0.1	NiFe (B3R2-1)	CoP (Z1192-5)
Composition	pure Ni	1-10% Fe in Ni	1-2%P in Co
Maturity	ASME-apprv'd	Dem/Val	Dem/Val
Grain Size (nm)	~100	<20	<20
Yield Strength (MPa)	670	1343	1485
Yield Strength (ksi)	97	195	215
Tensile Strength (MPa)	860	1750	1944
Tensile Strength (ksi)	130	254	282
Young's Modulus (GPa)	200	180	130
Hardness (HVN)	320	530	540
Elongation to Failure (%)	17	8.3	8.9
Elastic Limit (%)	0.33	0.54-0.78	1.2
Fracture Toughness	n/a	n/a	n/a
Density (g/cm ³)	8.9	n/a	8.7
Internal Stress, MPa (+), tensile, (-) compressive	n/a	+5 to -50	+70 to +100

Table 3 – main mechanical parameters of the materials as provided by Integran Technologies

5 Results of PICPRO® simulations

The extensive survey consists of 12 simulations involving CA of 2 different wall thicknesses and axial lengths. The values are reported in Table 4.

Four CA geometries have been investigated:

- CA of 500mm length and 3mm thickness (1 layer)
- CA of 1000mm length and 3mm thickness (1 layer)
- CA of 500mm length and 6mm thickness (2 layers)
- CA of 1000mm length and 6mm thickness (2 layers)

The capability of each device has been investigated by simulating 3 different crack speeds representative of a wide range of occurrences: 200, 275 and 350m/s.

CA MATERIAL	CA TYPOLOGY	CA THICKNESS (NO. OF LAYERS)	CA LENGTH	FRACTURE SPEED	REF
CoP nanometal alloy	Tight Sleeve arrestor. Obtained by wrapping a nanocrystalline ribbon around the main pipe	3mm (1)	500mm	200m/s	§5.1
				275m/s	
				350m/s	
		3mm (1)	1000mm	200m/s	§5.2
				275m/s	
				350m/s	
		6mm (2)	500mm	200m/s	§5.3
				275m/s	
				350m/s	
		6mm (2)	1000mm	200m/s	§5.4
				275m/s	
				350m/s	

Table 4 – CA geometrical parameters for simulations.

The following sections present the results of simulations performed on each crack arrestor. The fracture speed trend along the pipe and arrestor is plotted and details of each input parameters as well as most relevant outcomes are given nearby.

In detail, the following values are provided (see Figure 4 for reference):

- *exit speed*: the speed of the crack while exiting from the device;
- *minimum speed*: fracture minimum speed achieved due to the CA slow-down action. It is given just whether crack propagation beyond the CA is observed, as reported in Figure 4, right box;
- *arrest length*: it refers to the axial distance of the fracture tip from the CA entering edge. Obviously, it is reported in cases of arrest only;

- *steady speed beyond CA*: it represents the speed regime achieved by the crack after leaving the CA. Since it implies a further propagation beyond the device, it is given only whether ineffectiveness of the arrestor is envisaged;
- *CA damaged length*: it is the axial length of the damaged arrestor portion, due to the fracture passage.

It is important to highlight that while experimental evidences have been observed [4,5,6], which confirm the code capability in properly simulating conventional arrestors on large-diameters/high-grade pipelines, no test has been performed on nanometal reinforcements, which may support the code predictions whether nanometal reinforcements are considered.

Finally, the correct approach for interpreting the numerical outcomes suggests to consider the results as indicative and not as exact predictions.

NOMENCLATURE

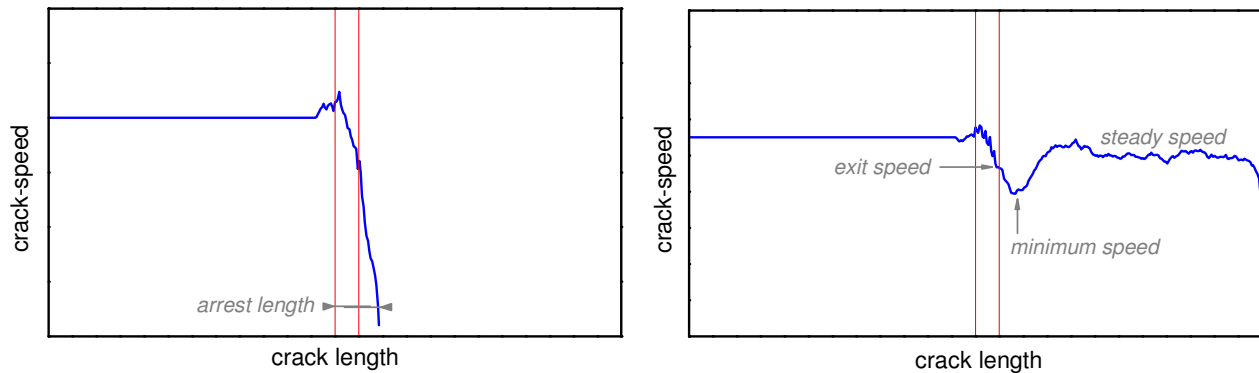


Figure 4 - nomenclature

5.1 3mm thick, 500mm long CA

Table 5 reports the results of the simulations performed on 3mm thick and 0.5m long arrestor. The device is predicted to be ineffective in arresting the crack propagating at speeds of 275m/s and 350m/s. Although a slow down occurs, it is not sufficient to assure an arrest and fracture further propagates beyond the CA at high speed.

A marked slow-down is expected whether initial speed is of 200m/s. In such a case an arrest is predicted to occur within a length exceeding the CA extent of ~0.4m, this fact being often not desirable. A conservative approach suggests to regard that occurrence as an “*uncertain case*”.

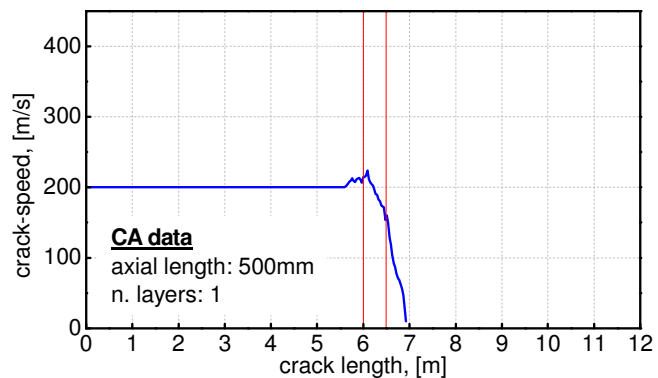
1 LAYER THICK, 500MM LONG ARRESTOR

Data

CA thickness	3mm (1 layer)
CA length	500mm
crack speed	200m/s

Outcomes

result	ARREST BEYOND CA
exit speed	150m/s
arrest length	0.9m from CA edge
CA damaged length	0mm

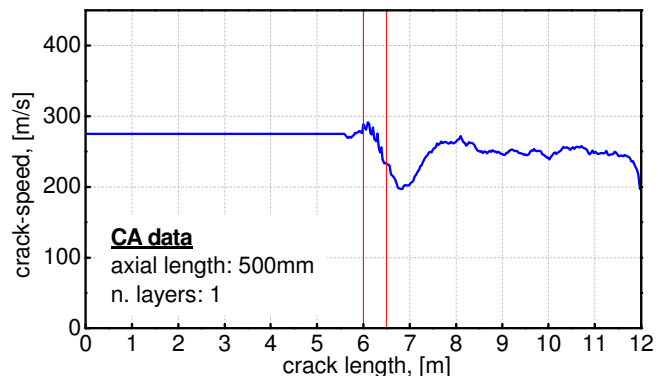


Data

CA thickness	3mm (1 layer)
CA length	500mm
crack speed	275m/s

Outcomes

result	PROPAGATION
exit speed	230m/s
minimum speed	195m/s
steady speed beyond CA	250m/s
CA damaged length	totally severed



Data

CA thickness	3mm (1 layer)
CA length	500mm
crack speed	350m/s

Outcomes

result	PROPAGATION
exit speed	320m/s
minimum speed	300m/s
steady speed beyond CA	345m/s
CA damaged length	totally severed

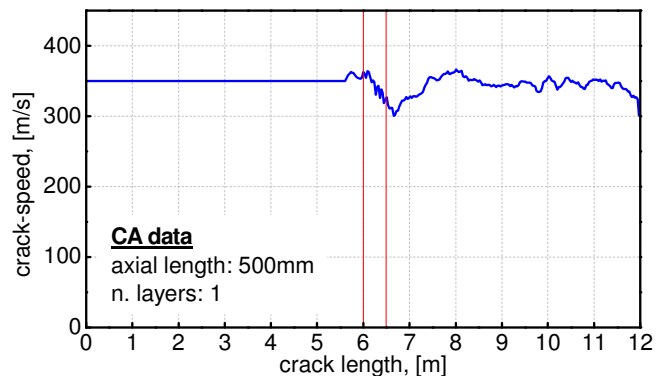


Table 5 – results of simulations on the 500mm long and 1 layer thick arrestor.

5.2 3mm thick, 1000mm long CA

In Table 6 the results are summarized which refer to a CA of analogous thickness of that simulated in §5.1 (3mm), but with an axial length of 1m. The increase of the axial length reflects on a better attitude of the device in arresting a fracture with speeds up to 275m/s. In this latter case an arrest shortly beyond CA is experienced, which however is conservatively considered as an *uncertain case* as explained in §3.2.

A special note has to be done for the simulation at 350m/s (lower box): an initial slow-down to ~300m/s occurs along the first half of the CA, followed by a propagation at constant speed along the second half. This indicates that the device is unable to supply the crack with an adequate constraint action and that a new steady regime of propagation has started, which keeps on going indefinitely along the CA.

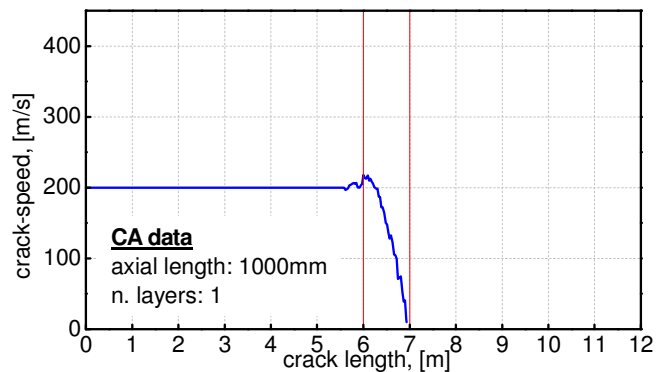
1 LAYER THICK, 1000MM LONG ARRESTOR

Data

CA thickness 3mm (1 layer)
CA length 1000mm
crack speed 200m/s

Outcomes

result **ARREST**
arrest length 0.95m from CA edge
CA damaged length 0mm

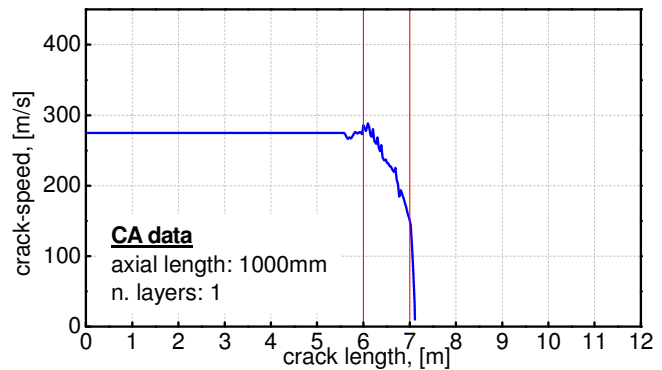


Data

CA thickness 3mm (1 layer)
CA length 1000mm
crack speed 275m/s

Outcomes

result **ARREST BEYOND CA**
exit speed 150m/s
arrest length 1.1m from CA edge
CA damaged length 100mm



Data

CA thickness 3mm (1 layer)
CA length 1000mm
crack speed 350m/s

Outcomes

result **PROPAGATION**
exit speed 315m/s
minimum speed 294m/s
steady speed beyond CA 340m/s
CA damaged length totally severed

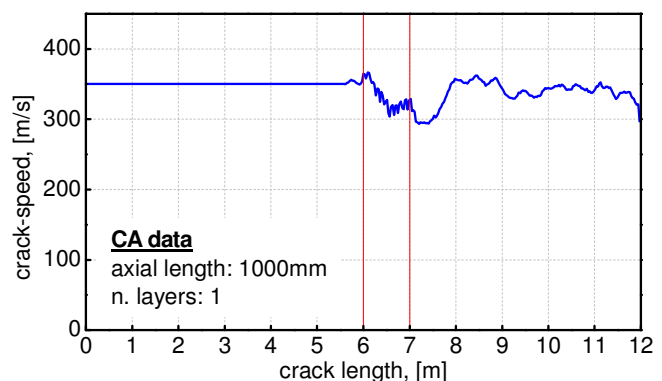


Table 6 – results of simulations on the 1000mm long and 1 layer thick arrestor.

5.3 6mm thick, 500mm long CA

Table 7 reports the results of the simulations performed on 6mm thick arrestor and 0.5m length. The CA is expected to arrest a fracture running with speeds of 200m/s and 275m/s. However, since an arrest beyond the device is expected for 275m/s, the outcome is regarded as an *uncertain case*, according to the motivations given in §3.2.

Whether a speed of 350m/s is considered, a relevant slow-down to ~60m/s is expected, followed by an acceleration up to a new steady state regime of 170m/s. Hence, the numerical outcomes consider it as a case of *fracture propagation*, that is *CA ineffectiveness*. However, crack propagation at speeds lower than about 80m/s are hardly probable from a practical point of view. At such low speeds instability phenomena have high probability to occur, which have large influences on the crack tip and may induce fracture redirection, spiralization and consequent arrest. Since representing an arrest/propagation border line event, it is considered as an *uncertain case*.

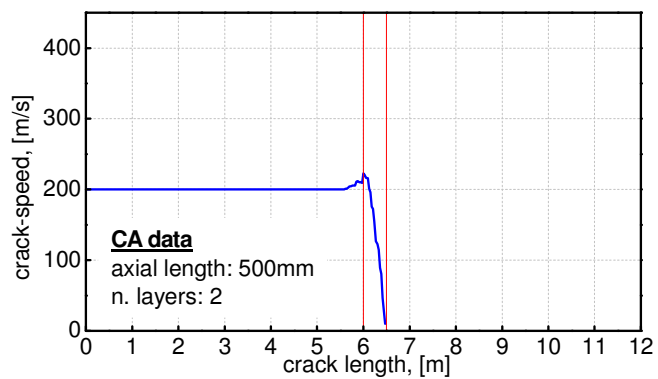
2 LAYERS THICK, 500MM LONG ARRESTOR

Data

CA thickness 6mm (2 layers)
CA length 500mm
crack speed 200m/s

Outcomes

result **ARREST**
arrest length 0.47 from CA edge
CA damaged length 0mm

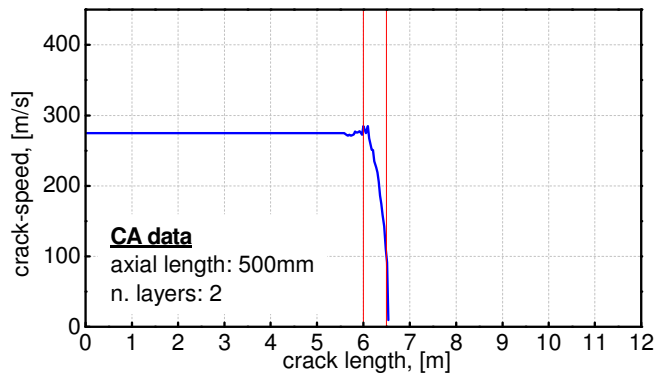


Data

CA thickness 6mm (2 layers)
CA length 500mm
crack speed 275m/s

Outcomes

result **ARREST BEYOND CA**
exit speed 100m/s
arrest length 0.55m from CA edge
CA damaged length 0mm



Data

CA thickness 6mm (2 layers)
CA length 500mm
crack speed 350m/s

Outcomes

result **POTENTIAL ARREST**
exit speed 265m/s
minimum speed 60m/s
steady speed beyond CA 170m/s
CA damaged length 0mm

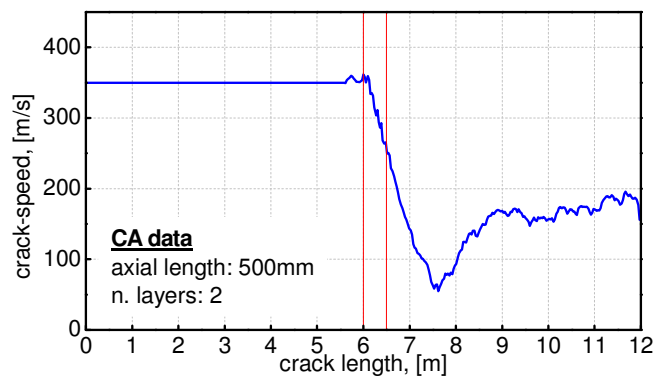


Table 7 – results of simulations on the 500mm long and 2 layers thick arrestor.

5.4 6mm thick, 1000mm long CA

The results of the simulations regarding the 6mm thick and 1m long arrestor are presented in Table 8. The device is expected to be effective in the whole range of speeds simulated. The fracture is rapidly slowed-down and arrested within the arrestor length.

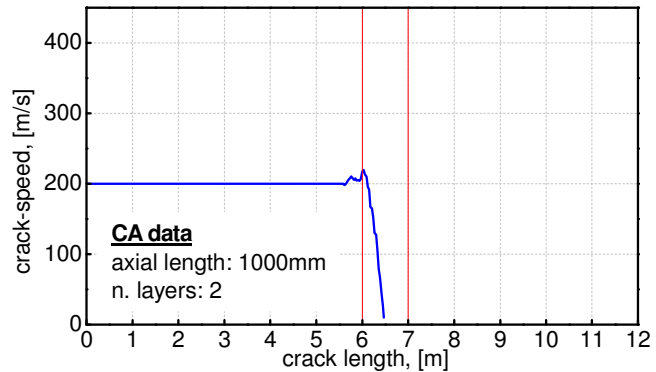
2 LAYERS THICK, 1000MM LONG ARRESTOR

Data

CA thickness 6mm (2 layers)
CA length 1000mm
crack speed 200m/s

Outcomes

result **ARREST**
arrest length 0.47m from CA edge
CA damaged length 0mm

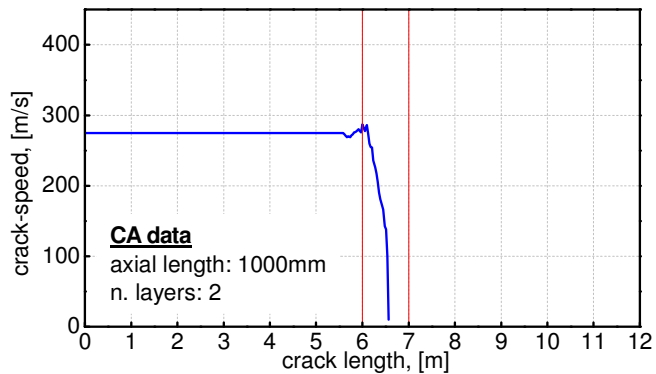


Data

CA thickness 6mm (2 layers)
CA length 1000mm
crack speed 275m/s

Outcomes

result **ARREST**
arrest length 0.57m from CA edge
CA damaged length 0mm



Data

CA thickness 6mm (2 layers)
CA length 1000mm
crack speed 350m/s

Outcomes

result **ARREST**
arrest length 0.81m from CA edge
CA damaged length 0mm

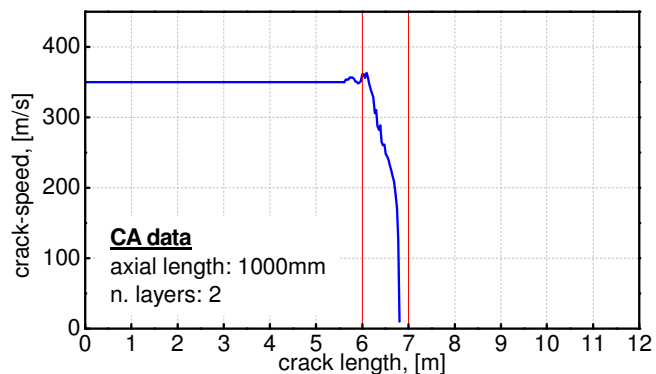


Table 8– results of simulations on the 1000mm long and 2 layers thick arrestor.

5.5 Resume of results

The results of the PICPRO[®] analysis are resumed in the plots Figure 5.

In the left box, the outcomes of the simulations regarding the 3mm thick arrestor are presented (see §5.1 and §5.2 for details). The diagram shows that the CA with an axial length of half meter reveals to be ineffective in arresting the propagating crack. An increase of the axial length up to 1m seems not to be decisive remedy, since the device capability is limited to crack speeds lower than 200m/s.

On the contrary, the increase of the CA thickness to 6mm produces significant improvements (see Figure 5 right box). The device appears to be able to lead the fracture to a rapid arrest, although some uncertainties remain for the half-meter long arrestor if high speeds are accounted.

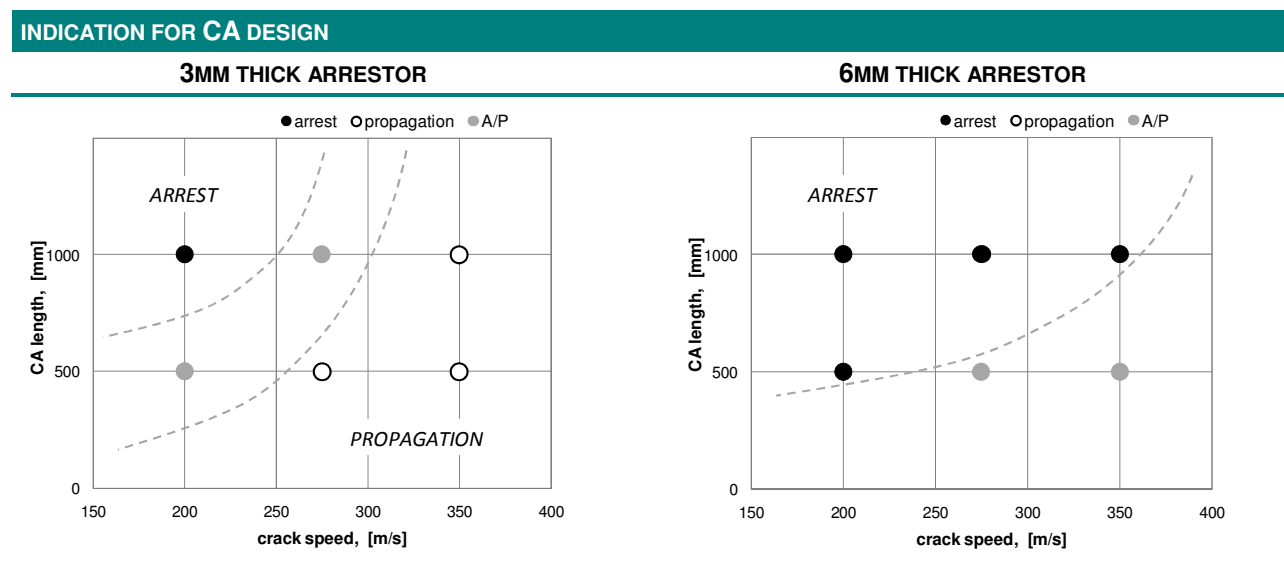


Figure 5 - FE simulation results of crack arrestor design

5.6 CoP to NiFe metal alloy comparison

An extra analysis has been performed by CSM on behalf of Integran Technologies, in order to compare the different behaviors of CAs made of NiFe and CoP nanometal alloy. The mechanical properties of such materials are reported in Table 3 and, as evident, they do not differ excessively each other, the CoP owning higher values of yield and tensile strengths, but lower Young's modulus than NiFe.

A limited number of simulations have been performed with a NiFe arrestor and the results compared with those relative to the corresponding CoP cases. The geometries of the NiFe arrestors involved in simulations are reported in Table 9.

CASE	CA THICKNESS (NO. OF LAYERS)	CA LENGTH	FRACTURE SPEED
#1	3mm (1)	500mm	275m/s
#2	6mm (2)	500mm	275m/s
#3	3mm (1)	1000mm	275m/s

Table 9 – CA geometrical parameters for simulations of CoP and NiFe CAs.

In Table 10 to Table 12 the results are briefly reported.

As indicated in case #1 Table 10, a CA of 500mm length and 3mm thickness exhibits the same ineffectiveness both in the case of CoP and NiFe nanometal alloy. At the same time, a thickness increase up to 2 layer (see Table 11) reflects in an improvement for both materials, which exhibit an arrest beyond CA.

According to those results it could be argued that the two materials own an analogous effectiveness. However, the plots indicate that the attitude of the CoP in slowing down the fracture is slightly better than that of the NiFe, as indicated by the lower *minimum speed* in Table 10 and the *arrest length* in Table 11. In other words, despite the analogous outcomes of case#1 and case#2, the two materials may present different behaviors in some particular cases, as indicated by case #3. The resulting crack speed diagram is plotted in Table 12, where the CoP alloy is predicted to lead the crack to an arrest shortly beyond the device, while the NiFe only determines a temporary slow-down of crack, which further propagates steadily at high speed.

The obtained results indicate that the capability of the NiFe material is predicted not to be very different from that of CoP. However, extrapolating the effectiveness of the nickel alloy from the CoP results is not allowed, since particular circumstances may be experienced where the attitude of the device is strictly connected to the material used.

Concluding, though appearing a valid alternative to CoP, the use of NiFe alloy in CA design requires a dedicated and extensive FE analysis in order to provide more detailed indications.

CoP TO NiFe ALLOY COMPARISON – #1

Data

CA thickness	3mm (1 layer)
CA length	500mm
crack speed	275m/s

Outcomes

	CoP	NiFe
result	PROP.	PROP.
exit speed	230m/s	235m/s
minimum speed	195m/s	215m/s
steady speed beyond CA	250m/s	260m/s
CA damaged length	tot. sev.	tot. sev.

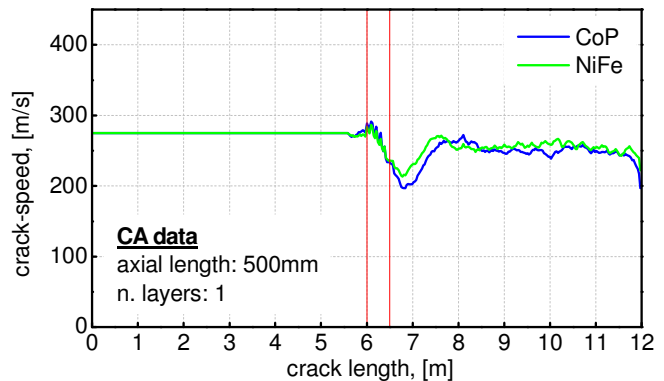


Table 10 – CoP to NiFe alloy comparison – results of case #1

CoP TO NiFe ALLOY COMPARISON – #2

Data

CA thickness	6mm (2 layers)
CA length	500mm
crack speed	275m/s

Outcomes

	CoP	NiFe
result	ARREST BEYOND CA	ARREST BEYOND CA
exit speed	100m/s	130m/s
arrest length	0.55m	0.60m
CA damaged length	0mm	0mm

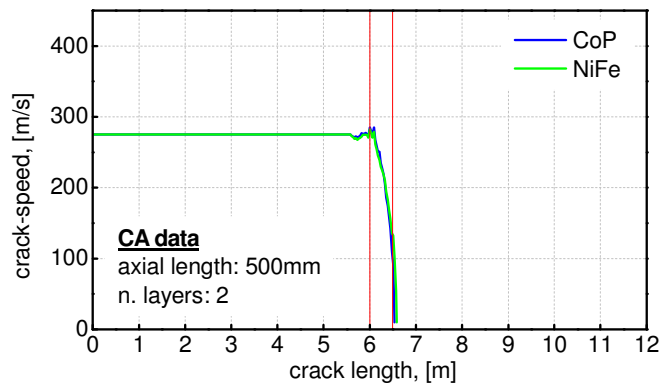


Table 11 – CoP to NiFe alloy comparison – results of case #2

CoP TO NiFe ALLOY COMPARISON – #3

Data

CA thickness	3mm (1 layers)
CA length	1000mm
crack speed	275m/s

Outcomes

	CoP	NiFe
result	ARREST BEYOND CA	PROP.
exit speed	150m/s	200m/s
arrest length	1.1m	-
CA damaged length	100mm	tot. sev.
minimum speed	-	140m/s
steady speed beyond CA	-	215m/s

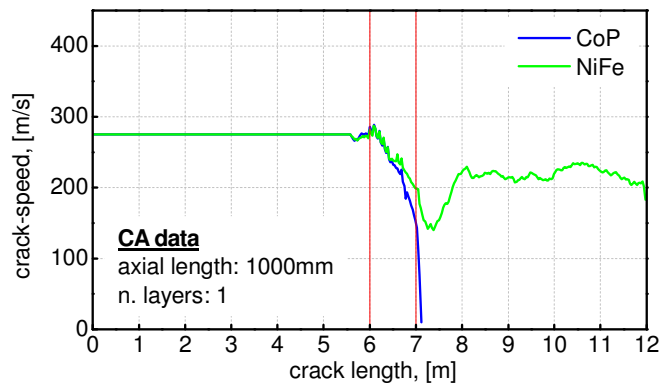


Table 12 – CoP to NiFe alloy comparison – results of case #3

6 Discussion and conclusion

In order to investigate the feasibility of adopting nanostructured metal alloys for constructing high performance crack arrestors (CA) for arresting ductile fracture propagation in high grade, large diameter pressurized gas pipeline, Integran Technologies asked CSM to undertake a Finite Element survey to furnish predictive indications on the effectiveness of such devices.

Simulations have been carried out by means of the CSM's code PICPRO[®] and involved a selected number of geometries, involving:

- 2 levels of CA wall thickness: 3 and 6mm,
- 2 levels of CA axial length: 0.5 and 1.0m,
- 3 levels of crack speed: 200, 275 and 350m/s.

A total number of 12 cases has been considered.

Concerning the material, Integran Technologies proposed three alternatives: a pure Ni, a NiFe alloy and a CoP alloy. Among those, the cobalt-based nanometal has been preferred, which exhibits both very high tensile properties (in terms of yield and tensile strength) and considerable ductility (high elongation to failure).

On the basis of the results presented in §5, some considerations can be done:

- ✓ Unsatisfactory attitude of arresting the crack has been exhibited by the 3mm thick arrestor. The half meter long device appears to be ineffective and non-conservative results are expected even though the axial length is increased to 1m;
- ✓ Beneficial effects are envisaged if the CA thickness is increased to 6mm. In such a case, the 0.5m long device shows good capability, except for very high crack speeds (350m/s). An increased axial length of 1m provides significant improvements within the whole crack speed range.

It is noteworthy to mention that the accuracy in determining the suitable CA geometry is correlated to the number of simulations performed and geometries investigated. The thickening of the range of explored metrics and crack speeds provides with more detailed information and improves the design optimization procedure.

In addition to the so far discussed survey, Integran Technologies asked CSM to perform an extra analysis, in order to provide some indication about the different capability of CAs made of NiFe and CoP nanometal alloy. The mechanical properties of such materials do not differ excessively each other, the CoP owning higher values of yield and tensile strengths, but lower Young's modulus than NiFe.

A limited number of simulations have been performed considering NiFe material and the results compared with those relative to the corresponding CoP cases.

In details, 3 metrics have been considered:

- a 3mm thick, 0.5m long arrestor;
- a 6mm thick, 0.5m long arrestor;
- a 3mm thick, 1.0m long arrestor.

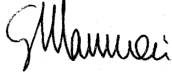
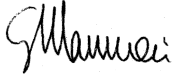
All devices were tested under a crack speed of 275m/s.

As result, although a slight supremacy of the CoP alloy has been observed, the NiFe behavior is expected to be not very inferior. Nevertheless, predictions obtained for one material cannot be considered valid for the other, since particular cases are envisaged where the attitude of the device is strictly connected to the adopted material. Concluding, though appearing a valid alternative to CoP, the use of NiFe alloy in CA design requires specific and extensive FE analysis in order to provide more detailed indications.

Lastly, it has to be highlighted that experimental evidences confirmed the code capability in properly simulating conventional arrestors on large-diameters/high-grade pipelines, [4,5,6], while no validating test has been carried out so far supporting the code predictions whether nanometal reinforcements are considered. Within this job PICPRO[®] has been applied to nano-metal alloys for the first time and validating experimental activities are thus recommended to verify and strengthen the PICPRO[®] predictive indications.

References

1. Berardo, G., Salvini, P., Mannucci, G., Demofonti, G. "On Longitudinal Propagation of a Ductile Fracture in a Gas Line Pipe: Numerical and Experimental Analysis" Intern. Pipeline Conference IPC2000, Calgary, October 2000.
2. Fonzo, A., Salvini, P., Di Biagio, M., Mannucci, G. "Full history burst test through finite element analysis", Proc. of the 4th Inter. Pipeline Conf., Calgary 2002, paper n. IPC2002-27120.
3. Salvini, P., Berardo, G., Demofonti, G., Mannucci, G. "Numerical model of the backfill constraint effect during fracture propagation on buried large diameter gas pipelines", Pipeline Technology Conference 2000, Brugge 22-24 May 2000.
4. Mannucci, G., Di Biagio, M., Demofonti, G., Fonzo, A., Salvini, P., Edwards, A. "Crack arrestor design by finite element analysis for X100 gas transportation pipeline", Proceedings of the 4th International Conference on Pipeline Technology, 9-13 May 2004, Ostend, Belgium.
5. Demofonti, G., et al., 2004, "Fracture propagation resistance evaluation of X100 TMCP steel pipes for high pressure gas transportation pipelines using full-scale burst tests", Proc. of the 4th International Conference on Pipeline Technology, Ostend, Belgium.
6. Fonzo, A., Meleddu, A., Di Biagio, M., Mannucci, G., Demofonti, G., Petersen, C.W., Biery, E. B., "Crack propagation modeling and crack arrestor design for X120", Proceedings of the 6th International Pipeline Conference, September 25-29, 2006, Calgary, Alberta, Canada.
7. Demofonti, G., Buzzichelli, G., Venzi, S., Kanninen, M.F., "Step by step procedure for the two specimen CTOA test", Proceedings of the 2nd International Pipeline Technology Conference, Ostend, Belgium, 1995.
8. P. Salvini, A. Fonzo, G. Mannucci, "Identification of CTOA and Fracture Process Parameters by Drop Weight Test and Finite Element Simulation", Engineering Fracture Mechanics, 70/3-4, pp. 553-566, 2003.
9. ISO 3183:2007 (E) "Petroleum and natural gas industries – Steel pipe for pipeline transportation systems", 2007.

COMMESSA N. S C140 005 <i>Internal order no.</i>		Progressivo per commessa: <i>Internal order serial number:</i> <input type="checkbox"/> avanzamento <i>Progress report</i> N. <input checked="" type="checkbox"/> finale <i>Final Report</i>
RAPPORTO N. 13685R <i>Report no.</i>		
RELATORI: <i>Authors</i> A.Meleddu A. Fonzo		
CAPO COMMESSA: <i>Job manager</i> G. Mannucci 		
RESPONSABILE CENTRO DI COMPETENZA: <i>Head of competence unit</i> G. Mannucci 		
RESPONSABILE AREA DI COMPETENZA (solo per il rapporto finale): <i>Head of competence area (only for final report)</i> M. Pontremoli (<i>ad-interim</i>)		
PRECEDENTI RIFERIMENTI SULLA COMMESSA: <i>Former internal order references</i>		
Rapporto N. <i>Report no.</i>	Data <i>Date</i>	Commessa N. <i>Internal order no.</i>